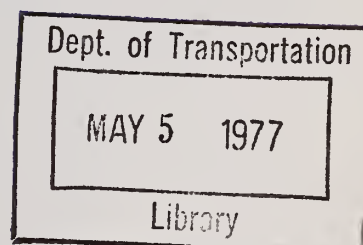


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DEVELOPMENT AND FIELD EVALUATION OF A TECHNIQUE FOR POLYMER IMPREGNATION OF NEW CONCRETE BRIDGE DECK SURFACES



September 1976
Final Report

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16. Abstract The development and field evaluation of a technique of impregnating new concrete bridge decks with an acrylic polymer system to a depth of 1 inch (25 mm) or more for protection against chloride induced corrosion of the reinforcing steel and freeze-thaw deterioration is reported. The durability of concrete treated with the polymer impregnation technique was determined in chloride intrusion, freeze-thaw and skid and abrasion resistance tests. The first polymer impregnation of the entire riding surface of a full size bridge deck was performed as a part of this research and development program in October 1974. A detailed discussion of this first field treatment and a general discussion of two subsequent full size bridge treatments which resulted in refinements to the treatment technique are included.			
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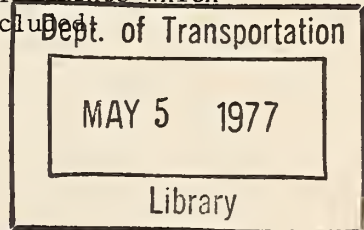


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PREFACE

One of the major problems facing the highway industry is chloride induced corrosion of concrete reinforcing steel and the subsequent deterioration of concrete bridge decks. The Federal Highway Administration's Office of Research and Development considers elimination of bridge deck deterioration as one of its highest priority efforts. The problems and the approach to solution of the problems are defined in Project 4B of the Federally Coordinated Program of Research and Development in Highway Transportation under the title, "Eliminate Premature Deterioration of Portland Cement Concrete." This report is a work unit in Task 1, "Elimination of Spalling and Delamination of Structural Concrete."

Assisting the author in the performance of this project was F. E. Causey, D. O. Arney, M. Northcutt, and other members of the Bureau of Reclamation Polymer Concrete and Structural Section under the supervision of G. W. DePuy, Supervisor, Unit 2 and C. E. Selander, Head, Polymer Concrete and Structural Section.

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DEFINITION OF TERMS PERTAINING TO POLYMER IMPREGNATION

<u>Term</u>	<u>Simple definition</u>
Polymer-impregnated concrete (PIC)	Hydrated portland cement concrete fully impregnated with a monomer system that is subsequently polymerized in situ.
Surface-impregnated concrete	A modification of PIC whereby the concrete is impregnated with polymer to some depth less than the full thickness of the specimen.
Monomer	The simplest basic molecule of a material that can be combined to form large molecules of a stable material (example - methyl methacrylate (MMA), which is in liquid form, is the monomer of poly (methyl methacrylate) polymer).
Monomer System	One or more monomers for use in PIC. Usually contains inhibitor and catalyst.
Polymer	A hardened (polymerized) plastic; the product of a large number of monomeric molecules reacting together.
Catalyst	A compound used to initiate the polymerization reaction.
Promoter	A chemical which is used to activate a catalyst to initiate polymerization of monomer.
Inhibitor	A chemical which prevents premature polymerization of monomer during shipping and storage.
Methyl methacrylate (MMA)	Monomers used in this study.
Styrene (S)	
Polyester (PE)	
Vinyl ester (VE)	
Trimethylolpropane trimethacrylate (TMPTMA)	
Butyl acrylate (BA)	

Drying	The removal of free water from concrete by heating.
Impregnation	The process of saturating hardened dried concrete with a monomer.
Polymerization	The reaction of monomer molecules joining together to form a polymer; for this study the change from liquid to solid state with plastics.
Polymer loading	The percent by weight of polymer in PIC.

1. INTRODUCTION

With the emphasis presently being placed upon highway safety and the adoption of a bare-pavement policy during the winter months, highway organizations have greatly increased their utilization of deicing chemicals on concrete bridge decks. A significant increase in the rate of concrete bridge deck deterioration has coincided with the increased use of deicing salts. Numerous studies have been performed to determine the cause of the deck deterioration with the resulting conclusions that chloride-induced corrosion of the bridge deck reinforcing steel leads to delamination and subsequent spalling of the upper zone of the bridge deck concrete.

Presently, the costs of repairing or replacing concrete bridge decks affected by this type of deterioration have become a substantial portion of the highway maintenance budgets of a number of states.

Responding to this problem, the Federal Highway Administration has initiated and sponsored various research activities directed towards reducing or eliminating this type of deterioration.

In 1966, the Bureau of Reclamation began investigation and development of concrete-polymer materials at its Denver Engineering and Research Center. These materials exhibited remarkable strength and durability and appeared to have potential applications in many areas of construction. Relative to the needs of the highway industry, polymer impregnated concrete (PIC) showed outstanding resistance to freeze-thaw type deterioration due to its low water permeability. Other studies showed that some types of PIC were also resistant to the types of corrosion experienced in saline water desalting plants.

In 1971, the Federal Highway Administration sponsored an initial research study at the Bureau to determine if it was feasible to increase the durability of new concrete bridge decks by impregnating the deck to 1 inch (25 mm) below the riding surface with an acrylic polymer. Since the Bureau builds bridges and other large concrete structures that could be improved by increasing durability there were direct benefits to the Bureau that could result from this study.

The results of the initial study showed that surface impregnation of concrete decks was technically feasible and the research agreement was modified to accomplish a full-scale research effort at the Bureau with the following objectives:

- To develop the process technology, materials, and equipment to surface impregnate new concrete bridge decks to a depth of 1 inch (25 mm) below the riding surface with polymer;

To design and construct the full-size equipment necessary to test the surface impregnation process in the field;

Use the equipment to demonstrate the process by surface impregnating the riding surface of a new bridge deck in the field;

Investigate the freeze-thaw performance and skid and wear resistance of concrete impregnated to a depth of 1 inch (25 mm) with polymer.

Periodic letter progress reports have been submitted to FHWA throughout this study. This report is the final report of the research activities of this effort.

2. SUMMARY AND CONCLUSIONS

A process of protecting new concrete bridge decks from chloride-induced rebar corrosion by impregnating the concrete to a depth of 1 inch (25 mm) with an acrylic monomer system was developed and refined. The process is composed of three separate but inter-related steps:

1. Drying to remove moisture from the zone to be impregnated
2. Impregnating the concrete with a monomer system composed of 95 percent methyl methacrylate (MMA), 5 percent trimethylolpropane trimethacrylate (TMPTMA), and a suitable polymerization catalyst
3. Polymerizing the monomer in situ by a thermal catalytic polymerization technique

A series of process and materials development tests was performed with the following results:

Drying. - Drying to remove moisture from the concrete zone to be impregnated was found essential to obtain the desired polymer penetration. Undried concrete, even after several months exposure to the low relative humidity environment of the Denver Laboratories, would not accept monomer at an impregnation rate feasible for practical applications.

Several types of forced drying were investigated including the use of microwaves, high frequency radio waves, and several sources of applied heat. From these techniques, drying with hot forced air was selected for field use. The heat application requirements for drying with this technique were initially established as follows:

1. Raise the concrete surface temperature to 250°F (121°C) over a period of 8 to 12 hours
2. Maintain this surface temperature for a period of 60 hours to complete the drying cycle

These parameters were subsequently used in the field and found to be unnecessarily conservative. Through combined laboratory and full scale field tests the drying requirements were reduced to a 16- to 24-hour heat application cycle as follows:

1. Apply sufficient heat to raise the concrete surface temperature to 250°F to 265°F (121°C to 129°C) over an 8- to 12-hour period
2. Maintain this temperature range, $\pm 20^\circ\text{F}$ (11°C) for a period of 8 to 12 hours

Monomer system. - Monomer systems composed of various combinations of MMA, styrene, polyester, TMPTMA, and vinyl ester were tested to determine their suitability for use in the bridge treatment process.

Systems composed of MMA, MMA-TMPTMA, and vinyl ester-styrene were found to be capable of impregnating concrete to the desired depth. A system composed of 95 percent MMA and 5 percent TMPTMA was subsequently chosen for field applications.

A series of tests was performed using various polymerization catalysts with the MMA-TMPTMA monomer system selected for field applications and with the other monomer systems listed above. Two azo-type catalysts, azobis-isobutyronitrile (AIBN) and azobis-dimethylvaleronitrile (AMVN) provided acceptable results with the MMA-TMPTMA monomer system. AMVN was ultimately selected for field use because of its superior performance over AIBN in initiating polymerization before monomer evaporation losses became detrimental.

Impregnation technique. - Four monomer impregnation techniques were evaluated. These included simple ponding and soaking with monomer, vacuum and vacuum-pressure variations of ponding, and the use of sand as a reservoir to hold monomer on the concrete surface until it penetrated the concrete to the desired depth. The use of vacuum and vacuum in combination with air pressure decreased the time required for monomer penetration but was judged impractical for field use unless monomer penetrations deeper than 1 to 2 inches (25 to 51 mm) were required. The method of impregnation which was selected for field use consists of spreading a layer of sand over the dried concrete surface, saturating the sand with monomer, and allowing the monomer to diffuse from the sand layer into the concrete surface. Using this technique a minimum of 4 to 6 hours was required for the monomer to penetrate to the 1-inch (25-mm) depth. A total monomer application rate of 1.2 to 1.3 pounds of monomer per square foot of treated concrete surface (5.8 to 6.4 kg/m²) was found to be adequate to provide the necessary penetration.

Due to the monomer-catalyst system's sensitivity to solar radiation it was found necessary to perform the impregnation cycle between sunset and sunrise or to shield the concrete from direct and indirect sunlight during the cycle.

Polymerization. - In situ polymerization of the impregnated concrete by both promoter-catalyst and thermal catalytic techniques was investigated. The tests with promoter-catalyst systems were generally unsuccessful. Thermal catalytic polymerization with hot air, hot water, steam, and combinations of these were tested for use under field conditions.

The technique of using hot forced air for polymerization was selected primarily because the same equipment used for the hot air drying cycle could thus be used for polymerization. The requirements of hot air polymerization of concrete impregnated with the 95-5 MMA-TMPTMA monomer system containing AMVN catalyst, assuring the monomer has penetrated the concrete to a depth of 1 to 2 inches (25 to 51 mm) are as follows:

1. Raise the treated concrete surface temperature to 160° to 180°F (71° to 82°C) over a 3-hour period
2. Maintain this temperature range for a minimum of 6 hours to complete the polymerization reaction

Durability Test

A series of durability tests was performed on concrete specimens impregnated with the surface treatment process to confirm that the treatment process provided the desired resistance to chloride ion intrusion.

In one test conducted at the FHWA Fairbank Highway Research Station Laboratories, treated concrete was subjected to over 600 daily applications of a 3 percent NaCl solution. During this test chloride concentrations at depths 1/2 inch (13 mm) below the surface remained far below that established as the corrosion threshold level. In freeze-thaw tests, reinforced concrete slabs were exposed to 69 weekly cycles of freezing, thawing with sodium chloride, and drying to determine if the impregnated zone would separate or delaminate from the lower unimpregnated concrete due to the slight difference in thermal expansion coefficients of the two zones. No such delamination occurred but the untreated concrete zone experienced severe spalling on the exposed sides while no deterioration was detected on the upper portion of the sides protected by impregnation. The top surface of the untreated concrete was deteriorated to a depth of 3/8 inch (9.5 mm) while only the surface laitance was lost from the surface of the treated specimen.

To determine the effects of the surface impregnation treatment on the abrasion and skid resistance properties of concrete, circular track tests were performed on treated and untreated concrete with three types of surface finish. This test involved over 1 million wheel passes. The results of this test showed that while no real increase in abrasion resistance was associated with polymer impregnation, neither did the treated concrete exhibit a propensity to polish or develop less skid resistance than conventional concrete.

Field Tests

Moving from the laboratory a series of surface impregnation treatments was performed in the field to evaluate and demonstrate the treatment process.

The first of these treatments was performed on a 10- by 14-foot (3.0- by 4.3-m) portion of a concrete slab on grade. This test was followed by the treatment of one 12- by 30-foot (3.6- by 9.1-m) lane of a small bridge located on the Denver Federal Center.

In both of these tests, the desired polymer penetrations were achieved showing that the treatment process was adaptable to field conditions. In October of 1974, the first full-scale surface impregnation of the entire riding surface of a bridge deck was performed by the Bureau on a bridge located in Cherry Hills Village near Denver. The treated surface was approximately 30 feet (9.1 m) wide by 60 feet (18.3 m) long. The treatment process involved 72 hours of heat application to dry the concrete followed by 24 hours of cooling. The impregnation cycle was performed during an overnight period of 14 to 16 hours utilizing a total monomer application rate of 1.7 lb/ft² (8.3 kg/m²) applied in three separate applications. Polymerization heating occupied about 18 hours. Test cores drilled from the treated surface at completion of the polymerization cycle showed polymer penetrations of 1 to 1-1/2 inches (25 to 38 mm) into the concrete.

Shortly after the surface impregnation treatment of this bridge the presence of roughly circular pattern cracking was noticed on the surface of the deck. These cracks were found to extend 2 to 3-1/2 inches (51 to 89 mm) into the concrete and though the cracks contained polymer they were not sealed with polymer. Physical and chemical analyses of the concrete indicated that there were no unusual detrimental chemical reactions in the specimens and that the concrete appeared sound. It was not possible to determine when the cracking occurred or the cause of the cracking. Reinspection of the two previous field treatments failed to reveal clear examples of similar cracking.

The surface impregnation process was subsequently used to treat two additional bridges. This work was performed in an implementation program sponsored by FHWA with the Bureau participating as technical advisor and will be reported in detail in a separate report. As a result of this work, however, the treatment process was refined to include a 16- to 24-hour drying heat cycle, a reduction in the monomer application rate from 1.5 to 1.7 lb/ft² (7.3 to 8.3 kg/m²) to 1.2 to 1.3 lb/ft² (5.9 to 6.4 kg/m²) and a reduction

in number of monomer applications from three to one. It was also learned that the surface pattern cracking occurred during the drying cycle, probably as a result of drying shrinkage. Future tests are planned to investigate techniques of eliminating this problem.

An interim report 6/ describing the surface treatment process, procedures, and materials used on the Denver bridge was prepared by the Bureau and published by FHWA in June 1975.

3. PROCESS DEVELOPMENT AND MATERIALS

The development of process technology and materials for surface impregnation began with the investigation of monomer systems and as various tests were performed, the investigation branched into four broad but related areas. Basically these study areas are directly related to the steps in the surface treatment process. The monomer system must be capable of penetrating bridge deck concrete. The concrete must be dried to remove free moisture from the zone to be impregnated. An impregnation technique which allows the monomer to penetrate the concrete to the desired depth within the allowable time must be used, and finally the monomer within the concrete must be polymerized to form the desired composite concrete-polymer material. Thus, the studies of the process and materials development program can be reported as:

1. Monomer screening tests and selection
2. Concrete drying studies
3. Impregnation studies
4. Polymerization studies

It should be understood, however, that the surface impregnation-polymerization process is composed of a series of interrelated, interdependent steps. As a result of these relationships the research and development program did not necessarily proceed in the chronological order that could be inferred from the organization of this report. That is, the concrete drying studies did not follow the monomer screening tests nor necessarily precede the impregnation studies. Instead, tests and studies were performed simultaneously in numerous areas of the process technology and the knowledge and experience gained in one study topic were used to modify or change the ongoing studies of the others.

3.1 Monomer Screening and Selection

From past experience it was known that two monomers, MMA and styrene (S), could be used to impregnate concrete. Each of these monomers alone, however, had drawbacks for bridge deck application. Modifications or other monomer systems would be needed.

Several monomer system characteristics were considered to evaluate the materials.

First, the monomer should have a low viscosity such that it could penetrate concrete to a depth of 1 inch (25 mm) within a reasonable time.

Second, the monomer should readily polymerize in the concrete, preferably by chemical means without the addition of energy.

Third, the resulting polymer should provide the desired improvement in freeze-thaw and chloride ion intrusion durability.

In addition to these major performance objectives, consideration was also given to other properties of monomer systems that might create safety hazards unique to a field application. Highly toxic monomers were eliminated from consideration as were those that were chemically reactive with concrete. Consideration was also given to the flammability characteristics of monomer candidates.

Using these broad objectives a series of monomer system screening tests was performed. It was learned during previous studies that monomer systems with viscosities greater than 5 to 10 cP were difficult to use for concrete impregnation. A series of Brookfield viscosity measurements was made on pure monomers and on various mixtures of monomers. Table 1 lists the monomer systems and the viscosities obtained from these measurements.

A number of the monomers and monomer mixtures had viscosities low enough to be potential candidates for concrete impregnation. To further evaluate their potential, a series of tests was performed to evaluate relative depths of concrete impregnation. In these tests a small impregnator built for use in manufacturing PIC was used to treat 3-inch (76-mm) diameter by 6-inch (152-mm) long concrete cylinders as follows:

1. The cylinders were oven-dried at 325°F (163°C) to constant weight. Normally this required an overnight drying cycle.
2. The oven-dried specimens were placed in 3-1/16-inch (78-mm) diameter stainless steel containers with open tops.
3. Sufficient monomer, catalyzed with 1 percent by weight azobiz-isobutyronitrile (AIBN) was poured into the containers to inundate the concrete and provide excess monomer coverage after impregnation.
4. To impregnate, the monomer-filled units were placed into the impregnator where they were exposed to various vacuum and soak cycles.
5. Following impregnation, the specimens were removed from the impregnator and the stainless steel containers and wrapped in aluminum foil for polymerization.
6. Polymerization was accomplished in an oven at 158°F (70°C). When cool the specimens were cut with a diamond saw to show the depth of impregnation.

Table 1

BROOKFIELD VISCOSITY OF MONOMERS

Monomer	Viscosity cP	Temperature °F
MMA	0.8	74
S	1.0	79
BA	0.9	73
GR-941	912.0	78
GR-480	1,253.0	80
GR-28V	59.0	79
SMP	995.0	63
TMPTMA	57.5	66
D-470	142.0	68
10% GR-28V - 90% S	1.1	82
20% GR-28V - 80% S	1.5	79
25% GR-28V - 75% S	1.8	80
50% GR-28V - 50% S	4.5	81
10% GR-941 - 90% S	1.2	78
20% GR-941 - 80% S	1.8	79
25% GR-941 - 75% S	2.3	78
50% GR-941 - 50% S	11.5	78
10% GR-480 - 90% S	3.3	79
20% GR-480 - 80% S	3.7	78
25% GR-480 - 75% S	5.4	79
50% GR-480 - 50% S	15.7	79
50% GR-28V - 50% BA	4.9	74
10% SMP - 90% S	1.1	81
25% SMP - 75% S	2.2	81
30% SMP - 70% S	3.4	81
40% SMP - 60% S	6.2	81
10% SMP - 90% MMA	1.1	57
30% SMP - 70% MMA	2.4	63
40% SMP - 60% MMA	4.5	61

Table 1 - Continued

Monomer	Viscosity cP	Temperature °F
5% D-470 - 95% S	1.0	68
10% D-470 - 90% S	1.0	68
20% D-470 - 80% S	1.3	68
30% D-470 - 70% S	3.8	68
40% D-470 - 60% S	5.0	68
50% D-470 - 50% S	7.5	68
5% TMPTMA - 95% MMA	0.8	66
10% TMPTMA - 90% MMA	0.8	66
15% TMPTMA - 85% MMA	0.9	66
20% TMPTMA - 80% MMA	0.9	66
25% TMPTMA - 75% MMA	1.0	66
30% TMPTMA - 70% MMA	1.1	66

MMA - Methyl methacrylate.

S - Styrene.

BA - Butyl acrylate.

GR-941 - Polyester.

GR-480 - Polyester.

GR-28V - Polyester.

SMP - Synthetic marble polyester.

TMPTMA - Trimethylolpropane trimethacrylate.

D-470 - Vinyl ester system containing 45% S.

The results of these tests are shown in table 2. The depths of impregnation are listed as minimum and maximum values that occurred in each specimen.

Reviewing these data, it can be seen that the MMA and the MMA-polyester systems consistently penetrated the concrete deeper than the S and S-polyester systems. Consequently, a 2-foot (610-mm) square by 4-inch (102-mm) thick concrete slab was used to determine the penetration of MMA under simulated field conditions. In this test the slab was oven-dried to constant weight at 325°F (163°C) and cooled to room temperature. A cylindrical stainless steel ring, 1.5-foot (457-mm) diameter by 3 inches (76 mm) tall was then calked onto the top surface of the concrete slab, forming a reservoir. MMA catalyzed with 1 percent by weight AIBN was added to the reservoir until a depth of 1 inch (25 mm) was obtained. This was allowed to soak into the concrete for 4 hours. At the end of the soak cycle, the excess monomer and the steel ring were removed. The slab was then wrapped in aluminum foil and placed in an oven at 158°F (70°C) overnight for polymerization. After polymerization the slab was sawed in half and examined for polymer penetration. A faintly visible darker zone indicated that the MMA had penetrated the concrete to a depth of 1-3/4 inches (44 mm).

Infrared absorption spectrography confirmed the presence of polymer in the impregnated zone. Low concentrations of polymer were found also at depths of 2 and 2-1/2 inches (51 and 64 mm). With these promising results six additional slabs were impregnated using the ponding technique. A comonomer system of 90 percent MMA and 10 percent TMPTMA was used. The TMPTMA monomer acts to crosslink MMA, improving its resistance to the effects of various solvents such as gasoline and diesel fuel. The copolymer is also darker and more easily seen in concrete.

In these tests soak times of 1-3/4, 2, and 4 hours were used. Three of the slabs treated were not oven-dried. The test results are listed in table 3.

Penetrations of greater than 1 inch (25 mm) were achieved in all the dried specimens. The 4-hour soak time resulted in depths greater than those of the 2-hour soak time, and the increase in depth appeared to vary with the square root of time. Monomer penetration into undried concrete - containing approximately 5 percent water by weight - was very shallow and uneven.

From these results it appeared that an MMA-TMPTMA monomer system could be used to surface impregnate concrete bridge decks. The MMA-polyester and some of the S-polyester monomer systems also appeared to have promise. It was decided to further evaluate all

Table 2

DEPTH OF IMPREGNATION TESTS
3-inch-diameter by 6-inch-long cylinders

Spec. No.	Time at 23 inches Hg vacuum min	Soak time at atmos pressure min	Polymer loading % <u>1</u> /	Penetration min - max inches	Monomer system % by weight
1-1	30	30	3.6	0.35 - 0.80	90S-10SMP
1-2	30	30	4.3	0.35 - 0.70	90S-10SMP
1-3	30	30	3.3	0.30 - 0.50	75S-25SMP
1-4	30	30	3.3	0.35 - 0.65	75S-25SMP
1-5	30	30	3.3	0.35 - 0.55	70S-30SMP
1-7	30	30	2.2	0.15 - 0.40	60S-40SMP
1-8	30	30	2.4	0.10 - 0.35	60S-40SMP
1-9	30	30	2.0	0.05 - 0.40	50S-50SMP
1-10	30	30	2.0	0.10 - 0.35	50S-50SMP
2-1	15	45	3.4	0.40 - 0.75	75S-25SMP
2-2	15	45	3.4	0.40 - 0.75	75S-25SMP
2-3	15	45	3.6	0.40 - 0.95	70S-30SMP
2-4	15	45	3.3	0.35 - 0.60	70S-30SMP
2-5	15	45	2.5	0.20 - 0.45	60S-40SMP
2-6	15	45	2.8	0.25 - 0.60	60S-40SMP
6-1	15	45	3.9	1.00 - 1.10	90MMA-10SMP
6-2	15	45	4.4	0.90 - 1.20	90MMA-10SMP
6-3	15	45	3.2	0.70 - 0.80	70MMA-30SMP
6-4	15	45	3.4	0.70 - 0.90	70MMA-30SMP
6-5	15	45	3.3	0.60 - 0.80	60MMA-40SMP
6-6	15	45	2.8	0.60 - 0.70	60MMA-40SMP
4-1	20	90	4.2	0.40 - 0.65	75S-25SMP
4-2	20	90	4.0	0.25 - 0.75	75S-25SMP
4-4	20	90	4.5	0.35 - 0.75	70S-30SMP
4-5	20	90	3.5	0.40 - 1.10	70S-30SMP
4-7	20	90	4.5	0.25 - 0.55	90S-10SMP
4-8	20	90	4.7	0.30 - 0.80	90S-10SMP

Table 2 - Continued

Spec. No.	Time at 23 inches Hg vacuum min	Soak time at atmos pressure min	Polymer load %	Penetration min - max inches	Monomer system % by weight
5-1	20	90	5.5	1.50	90MMA-10SMP
5-2	20	90	5.3	1.50	90MMA-10SMP
5-3	20	90	3.8	0.70 - 1.00	70MMA-30SMP
5-4	20	90	4.1	1.00 - 1.10	70MMA-30SMP
5-5	20	90	3.1	0.50 - 0.70	60MMA-40SMP
5-6	20	90	3.7	0.70 - 1.00	60MMA-40SMP
3-1	15	45 @ 5 lb/in ²	3.1	0.40 - 0.70	75S-25SMP
3-2	15	45 @ 5 lb/in ²	3.2	0.25 - 0.65	75S-25SMP
3-3	15	45 @ 5 lb/in ²	2.5	0.20 - 0.70	70S-30SMP
3-4	15	45 @ 5 lb/in ²	3.5	0.40 - 0.95	70S-30SMP
3-5	15	45 @ 5 lb/in ²	3.0	0.25 - 0.70	60S-40SMP
3-6	15	45 @ 5 lb/in ²	2.6	0.25 - 0.65	60S-40SMP
2-7	15	45	4.1	0.65 - 1.05	100 MMA
2-8	15	45	4.1	0.85 - 1.10	100 MMA
2-9	15	45	3.8	0.35 - 0.80	100 S
2-10	15	45	3.5	0.35 - 1.00	100 S

S - Styrene.

SMP - Synthetic marble polyester.

MMA - Methyl methacrylate.

1/Percent polymer by total weight of concrete specimen.

Table 3

RESULTS OF POND TECHNIQUE ON CONCRETE SLABS

Slab size (ft ²)	Monomer system <u>1</u> /	Soak times hours	Oven dried	Depth of penetration inches
2	MMA	4	Yes	1-3/4
2	<u>2</u> / 90-10	4	Yes	1-3/4
2	90-10	1-3/4	Yes	1-1/8
2	90-10	1-3/4	No	1/2
2	90-10	1-3/4	No	1/4-1/2
4	90-10	2	Yes	1-1/4
4	90-10	2	No	1/8-1/4

1/ 1 percent AIBN used as catalyst.

2/ 90 percent MMA - 10 percent TMPTMA.

three systems during the impregnation and polymerization studies and to shift the emphasis in the monomer screening tests to evaluation of promoter-catalyst systems for use with the monomer systems.

Controllable ambient temperature polymerization is a highly desirable characteristic of a monomer system for surface impregnation applications. Normally, this is accomplished in the plastics industry by adding a promoter-catalyst system to the monomer. With this technique, the promoter activates the catalyst at relatively low temperatures causing it to generate free radicals which subsequently cause polymerization of the monomer.

To develop this technique for bridge deck applications, a series of catalyst-promoter tests was performed on polyester-styrene and polyester-MMA mixtures. In these tests, 100-gram samples of the monomer systems were polymerized at ambient room temperatures. During the tests the times to reach gel state, the time of final cure, and the general appearance of the resulting polymer were determined. The results are listed in tables 4 and 5. All of the polyester-styrene systems exhibited shrinkage fractures and surface flaking in the resulting polymer. The shrinkage fractures were expected and generally are inherent in bulk polymerization unless special precautions are taken to prevent them. The surface flaking, however, is associated with unpolymerized monomer within the polymer and has a weakening affect upon the material. The polyester-monomer systems that completely polymerized did not exhibit the surface flaking phenomena but the times required for final cure were quite long. Following these tests 26 additional polyester-monomer mixtures were tested using various catalyst-promoter systems with similar results. It was thus concluded that ambient temperature promoter-catalyst polymerization with polyester-monomer systems was not practical for bridge deck surface impregnations.

In similar tests with MMA and MMA-TMPTMA mixtures a promoter-catalyst system composed of di-methyl-p-toluidine and benzoyl peroxide gave uniform, repeatable bulk polymerization times of 2 to 30 minutes depending upon promoter-catalyst ratio and concentrations, and it was decided to attempt to impregnate several concrete slabs using this promoter-catalyst system for polymerization. In these tests the concrete slabs were oven-dried to constant weight at 325°F (163°C) and cooled to ambient room temperature. A 1-inch (25-mm) deep reservoir of monomer was placed over the concrete surface and allowed to soak into the concrete as previously described. The results of these tests are shown in table 6 and discussed in detail in section 3.4 of this report. Faced with these generally poor results, the decision was made to abandon the active search for a promoter-catalyst monomer system for bridge deck applications and concentrate instead on catalysts suitable for thermal-catalytic polymerization.

Table 4

ROOM TEMPERATURE POLYMERIZATION OF POLYESTER-STYRENE MIXTURES

Polyester	PE-S mixture	Catalyst percent MEKP	Promoter percent CN	Gel time	Cure time	Appearance of polymer
GR-28V	50-50	1	1	15 min	46 min	Badly fractured
	50-50	1	1/2	22 min	46 min	Fractured - some flaking
	50-50	1	1/4	25 min	44 min	Fractured - flaking
	50-50	1/2	1	31 min	ON	Deep surface flaking
	50-50	1/2	1/2	33 min	1 hour	1/2 weight lost to flaking
					36 min	
	50-50	1/2	1/4	42 min	ON +	Badly flaked
GR-94I	25-75	1	1	22 min	4 hours	Deep fractures - flaking
	25-75	1	1/2	42 min	ON	Surface fractures
	25-75	1/2	1	49 min	ON	One circumferential fracture
	25-75	1/2	1/2	49 min	ON	Deep circumferential fracture
	10-90	1	1	ON	2-3 days	Solid - no fractures
	50-50	1	1	19	ON	Quartering fractures - light flaking
	50-50	1	1/2	18	ON	Deep fractures - bottom flaking
	25-75	1	1	33	ON	Deep circumferential fractures
	25-75	1	1/2	35	ON	Badly flaked
	25-75	1/2	1/4	53	ON	Badly flaked

MEKP - Methyl ethyl ketone peroxide.

CN - Cobalt naphthenate.

ON - Overnight.

Table 5

ROOM TEMPERATURE POLYMERIZATION OF POLYESTER - MMA MIXTURES

Polyester	PE-MMA mixture	Catalyst	Promoter	Gel time	Cure time	Appearance of polymer
GR-28V	50-50	1 percent MEKP	1/2 percent CN	ON	1-1/2 days	Solid - ring fracture at top
	50-50	1 percent BP	1/2 percent DMA	1 hour - 33 min	ON	Vertical fracture
	50-50	1 percent MEKP	1/2 percent DMA	32 min	ON	Surface and vertical fracture
			1/2 percent CN			
	50-50	1/2 percent MEKP	1/2 percent DMA	X	X	
	50-50	1/2 percent BP	1/2 percent DMA	2 hours - 10 min	ON	Vertical fracture
	50-50	1/2 percent MEKP	1/4 percent DMA	1 hour - 13 min	2 days	Solid polymer
			1/2 percent CN			
	75-25	1 percent MEKP	1/2 percent CN	1 hour - 34 min	ON	Quartering fractures
	25-75	1/2 percent BP	1/2 percent DMA	X	X	
	25-75	1/2 percent MEKP	1/2 percent DMA	X	X	
	25-75	1/2 percent MEKP	1/4 percent DMA	4 hours	X	
			1/2 percent CN			
	10-90	1/2 percent MEKP	1/2 percent DMA	X	X	

MEKP - Methyl ethyl ketone peroxide.

CN - Cobalt naphthenate.

DMA - Dimethyl aniline.

BP - Benzoyl peroxide.

X - Did not gel/cure.

ON - Overnight.

Table 6

PROMOTER-CATALYST SYSTEMS FOR SURFACE IMPREGNATION
 Ponding Impregnation of Concrete Slabs
 4 Inches Thick

Monomer system	Catalyst (%)	Promoter (%)	Impregnation time (hours)	Results 1/
60 S - 40 SMP	2 - MEKP	1/2 - CN	-	Gelled after 40 minutes
90 MMA - 10 TMP/TMA	1 - BP	1/4 - DMT	1.75	No polymerization
90 MMA - 10 TMP/TMA	1 - BP	1/4 - DMT	-	Gelled after 45 minutes
90 MMA - 10 TMP/TMA	1 - BP	0.05 DMT	1.75	No polymerization
90 MMA - 10 TMP/TMA	1 - BP	0.07 DMT	-	Gelled in 65 minutes
90 MMA - 10 TMP/TMA	1 - BP	0.07 DMT	2.0	No polymerization
90 MMA - 10 TMP/TMA	1 - BP	0.25 DMA	2.0	No polymerization
95 MMA - 5 TMP/TMA	1 - BP	0.50 DMA	-	Gelled after 45 minutes

MEKP - Methyl ethyl ketone peroxide.

CN - Cobalt naphthenate.

BP - Benzoyl peroxide.

DMT - Dimethyl-para-toluidine.

DMA - Dimethyl aniline.

1/ None of these systems resulted in satisfactory concrete impregnation. The systems that gelled on the surface of the concrete did not penetrate deeper than 1/4 inch (6.4 mm). Those that did not polymerize were subsequently lost through evaporation. The penetration depths of the systems that did not polymerize were not determined. During these tests it was not uncommon to find identical monomer-catalyst-promoter systems that would gell on the surface of one slab and fail to polymerize on another.

Catalyst System

Benzoyl peroxide (BP) is probably the most widely used catalyst for polymerization of acrylic monomers. This material is efficient in that a high degree of conversion is usually obtained and it can activate the polymerization reaction at the relatively low temperatures of approximately 140° to 160°F (60° to 70°C). The early polymerizations at the Bureau were invariably accomplished with this chemical.

Certain hazards, however, can be associated with the use of BP. It is flammable and toxic. The dry forms of the chemical are shock sensitive and, when mixed with monomer in the presence of some of the impurities encountered in concrete, an unplanned promoter-catalyst type of polymerization is potentially possible.

Another problem that accompanied some of the early work with BP at the Bureau was the inability to achieve full polymerization of BP catalyzed MMA in concrete. Generally, this problem occurred in fully impregnated concrete specimens as evidenced by an unpolymerized monomer saturated core area in the center of certain specimens.

For these reasons BP was considered unsuitable for surface impregnation applications. The other types of peroxide catalysts such as methyl-ethyl-keytone-peroxide (MEKP) and lauryl peroxide were believed to have similar properties and after only preliminary screening considerations were judged less than suitable.

The azo-type catalysts have also been used at the Bureau with excellent results. Generally these materials, while more expensive than the common peroxides, demonstrate excellent stability in the presence of impurities, relatively low reaction temperatures, and essentially complete monomer-to-polymer conversion. The azo catalysts discussed herein are not shock sensitive, although most are classified as flammable and are, of course, heat sensitive.

The azo catalyst that had been used most extensively in the Bureau's PIC research program was α -t-butylazo isobutyronitrile. This chemical had performed remarkably well in numerous polymerization tests and small experimental quantities of MMA containing one-half percent by weight catalyst had remained stable for storage periods of over 1 year. This catalyst system, having a 10-hour half life at 174°F (79°C), has a somewhat higher initiation temperature than some of the other azo catalysts such as (AIBN) which has a 10-hour half life at 147°F (64°C).

Due to its lower half life temperature AIBN was the first azo material selected for use as a surface impregnation polymerization catalyst with the MMA monomer system. Numerous tests with this catalyst were performed while investigating the drying, impregnation, and polymerization requirements of the surface treatment process. Initially the catalyst was believed to perform quite well. As the process technology became more refined, however, it was apparent that some of the less consistent test results could be improved by a more reactive catalyst (lower 10-hour half-life temperature).

A vinyl polymerization catalyst (AMVN) with a 10-hour half-life at 126°F (52°C) was tested in comparison to AIBN. Ten-gram samples of monomer (90 percent MMA - 10 percent TMPTMA) containing 1 percent AMVN were placed in 104°F (40°C), 113°F (45°C), and 122°F (50°C) water baths and the time to polymerization peak exotherm determined. These times were 92, 63, and 25 minutes, respectively. A similar test with 1 percent AIBN in monomer at 122°F (50°C) did not polymerize after 2-1/2 hours.

A 100-gram sample of 90 percent MMA - 10 percent TMPTMA was prepared with 1 percent AMVN and left in the dark at ambient room temperatures of 70° to 75°F (21° to 24°C) for 18 hours to determine if polymerization would be likely to occur prior to completion of an impregnation cycle. At the end of the 18th hour there was no visual evidence of polymerization and Brookfield viscosity measurements on the sample confirmed that no polymerization had occurred. Several 4- and 6-inch (102- and 152-mm) thick concrete slabs were then impregnated with the 90-10 monomer system containing 1 percent AMVN and polymerized with techniques being used at the time. The resulting polymer depths and uniformity were excellent.

Before a decision to switch to the AMVN catalyst was made the use of this material under field conditions was considered. Due to its lower 10-hour half life temperature AMVN can be stored only under refrigerated conditions at temperatures less than 50°F (10°C) and monomer containing AMVN should not be stored; thus, monomer-catalyst mixing need be done immediately before use. The catalyst is not shock sensitive but it is flammable and can be ignited with an open flame or continuous electric arc. AMVN and its decomposition products are of relatively low toxicity and are irritating to the eyes.

These various properties at first appeared detrimental to the choice of AMVN as a catalyst for field use. However, upon further consideration it was found that the normal safety procedures and safety factors prudently applied to the use of BP and AIBN were sufficient to provide ample safety provisions for the field use of AMVN. This material was thus selected for use. Numerous polymerization reactions both in the laboratory and in the field have proven AMVN to be a very reliable catalyst.

3.2 Drying Studies

Depending upon the climatic conditions and concrete composition, cured concrete bridge decks will generally contain from 2 to 6 percent free moisture based on weight. During the various programs performed to investigate and develop PIC it was found that this moisture interfered with the penetration of monomer into the concrete and that the resulting polymer loading varied inversely with concrete water content.

In the beginning of the surface impregnation program, tests were conducted to determine if the relatively shallow, 1 inch (25 mm), polymer penetration could be accomplished without the necessity of drying. These tests, however, gave poor results unless the moisture in the zone to be impregnated was removed. A series of tests designed to evaluate drying techniques, times, and temperatures was performed.

Drying Techniques

Various techniques are available for drying concrete for surface impregnation application. During this research program the following techniques were evaluated:

1. Microwave heating
2. Open flame (gas-fired torch)
3. Hot forced air (gas-fired space heaters - ovens)
4. Infrared (gas-fired heater)
5. Electro-osmosis
6. Radiofrequency

Two 3-inch (76-mm) diameter by 6-inch (152-mm) long concrete cylinders were dried to equilibrium weight in a microwave oven built for home use. One cylinder had been soaked in water for several days prior to drying. The second had been cured in a 50 percent relative humidity environment for approximately 1 month prior to drying.

These cylinders were placed separately in the microwave oven for 5-minute intervals, removed, weighed, and returned to the oven for continued drying until equilibrium weight was obtained.

The water-soaked cylinder required a drying cycle of 40 minutes (cylinder was out of the oven for 11 minutes of this cycle to permit weighing) to reach equilibrium weight. Water loss was 4.9 percent by weight and the concrete surface temperature was 390°F (199°C) at cycle completion.

The cylinder from the 50 percent relative humidity environment required a 35-minute drying cycle (during 6 minutes of the cycle this specimen was out of the oven for weighing) to reach equilibrium weight. Its water loss was 1.25 percent by weight and the concrete surface temperature at drying completion was 370°F (188°C).

To check the efficiency of the microwave drying, the cylinders were then placed in a 305°F (152°C) forced air oven and dried for 110 hours. No additional weight loss was experienced in either cylinder. Previous forced air drying tests performed during the PIC research program indicate that 8 and 20 hours were required to expel all free water from identical-sized specimens at 302°F (152°C) and 257°F (125°C), respectively. ^{1/} Thus, the microwave drying on these specimens was approximately 16 to 40 times faster than the forced air oven drying.

There are, however, several drawbacks to the field use of microwaves for this type of application. Microwave scatter in the vicinity of the bridge deck could pose safety problems. Although microwave drying is a highly efficient process, the electric power requirement for a field unit would still be quite large. Also, it was not known if microwave equipment of sufficient size to meet the drying requirement of bridge decks was commercially available. To resolve these potential problems 10 suppliers or manufacturers of microwave equipment were contacted. The consensus of opinion expressed by these organizations was that the desired equipment could be constructed and used for the purpose desired but that prototype equipment of this size would require a capital investment in the range of \$250,000 to \$500,000. This amount of money greatly exceeded that available in the program and the investigation of microwave drying techniques and equipment was abandoned.

The use of radiofrequency waves for concrete drying was considered briefly but even though the safety problems were not as severe as with microwaves the capitalized equipment cost was similar and the technique was abandoned for this reason. It is interesting to note that during a brief check of radiofrequency drying with a 10-kW unit at 40.68 MHz, the temperature of a 6-inch (152-mm) by 6-inch (152-mm) by 4-inch (102-mm) water-saturated concrete slab was raised from 75°F (24°C) to 200°F (93°C) in 3 minutes. An attempt to increase this temperature with an additional 1-1/2 minutes exposure in this high-powered equipment resulted in the slab exploding rather violently due to the generation of steam pressure within the concrete porosity.

A series of tests involving three types of gas-fired drying devices was then performed. An open flame from a torch similar to a weed burner was applied directly to the surface of concrete slabs until

the selected surface temperature was obtained for the desired time duration. A 300,000 Btu (87.9 kW) space heater with an electrically powered blower was used in a similar manner by directing its hot air onto the surfaces of concrete slabs. The third device, a 120,000-Btu (35.2-kW) gas-fired infrared heater designed for use as an asphalt pavement patcher was tested by supporting it over concrete test slabs. Figures 1, 2, 3, and 4 show these drying devices in operation.

The concrete slabs used in these tests were cured in a 100 percent relative humidity environment at 73°F (23°C) and were fully saturated at the beginning of drying. Following the various drying tests the slabs were cooled to surface temperatures of 110° to 120°F (43° to 49°C) and impregnated with the 90-10 monomer system using 1 percent by weight AIBN as catalyst and the 2-hour ponding impregnation technique. Polymerization of the monomer was accomplished by wrapping the slabs in polyethylene and placing them in a forced-air oven at 158°F (70°C). The test data, including drying temperature as measured on the surface of the slabs, drying times, and resulting polymer penetrations are shown in table 7.

The 2-foot (61-cm) by 2-foot (61-cm) by 4-inch (10-cm) slabs were small enough to be moved by hand. During polymerization they were thus placed directly on the floor of the forced-air oven where they received heat only on their top and side surfaces. The 3-foot (910-mm) square slabs, that were dried with the infrared heater, required a forklift for handling and during polymerization were placed on blocks in the oven to facilitate removal of the fork blades. In this arrangement they received heat on all surfaces. The 3/4-inch (19-mm) polymer penetration apparently resulted from the effects of bottom heat on the first three 3-foot (910-mm) square slabs treated. The last slab treated with the infrared heat source was placed flat on the polymerization oven floor and exhibited a polymer penetration of 1 inch (25 mm).

During these tests, data were kept on the amount of water removed and the amount of gas consumed in each test. Using these data and only the cost of the gas, which at that time was \$0.10 per pound (\$0.045 per kg), the following costs to remove water from the concrete were computed:

Space heater	\$0.48/lb (\$0.22/kg)
Open flame	\$0.33/lb (\$0.15/kg)
Infrared	\$0.23/lb (\$0.10/kg)

These tests indicated that adequate concrete drying could be accomplished by all three gas-fired drying techniques. The infrared method, however, appeared to be the most efficient and since larger infrared units were commercially available and readily adaptable to use on full-size bridge decks this technique was selected as a



Figure 1. - Drying test with open flame torch.

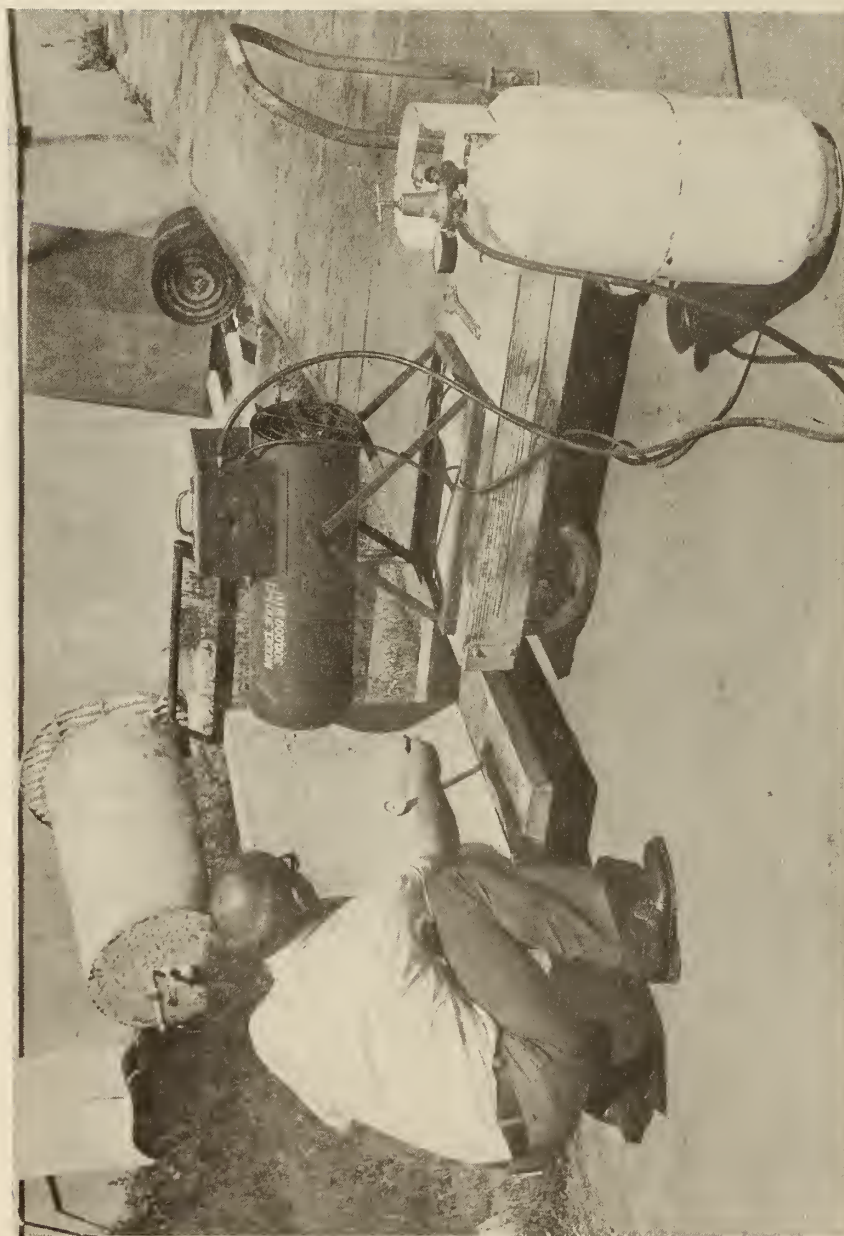


Figure 2. - Drying test with space heater.



Figure 3. - Drying test with gas infrared heater.



Figure 4. - Bottom view of infrared heater.

Table 7

RESULTS OF HEAT METHOD STUDIES FOR DRYING CONCRETE
FOR SURFACE IMPREGNATION

Specimen size ft x ft x in.	Drying method	Heating temperature and time	Polymer penetration in.
2 x 2 x 4	Open flame	450°F for 1 hour	1-1/4
2 x 2 x 4	Open flame	400°F for 1 hour	1-1/4
2 x 2 x 4	Open flame	350°F for 1 hour	1
2 x 2 x 4	Open flame	350°F for 1 hour	1-1/4 - irregular
2 x 2 x 4	Space heater	350°F for 1 hour	1-1/8
2 x 2 x 4	Space heater	300°F for 1 hour	1-1/8 - 1-1/4
2 x 2 x 4	Space heater	300°F for 1 hour	1
2 x 2 x 4	Space heater	290°F for 1 hour	1-1/4
2 x 2 x 4	Infrared	275°F for 1 hour	1
3 x 3 x 4	Infrared	300°F for 1 hour	*3/4
3 x 3 x 4	Infrared	300°F for 1-1/2 hours	*3/4
3 x 3 x 4	Infrared	300°F for 1-1/2 hours	*3/4
3 x 3 x 4	Infrared	300°F for 1-1/2 hours	1

* Placed on blocks during polymerization.

standard laboratory drying technique for the slabs subsequently used in the impregnation and polymerization tests. During these additional tests it was found that polymer penetrations of 1-1/4 inches (32 mm) to 1-1/2 inches (38 mm) could be accomplished with a drying time of 2 hours after the concrete surface reached a temperature of 300°F (149°C).

Several manufacturers of infrared equipment were contacted concerning the cost of large infrared heaters capable of heating a concrete bridge surface. From their responses it was learned that standard heater units could be purchased for approximately \$5,000. Electric infrared heaters were also considered but it was concluded that sufficient electric power to operate such a system would not commonly be available at bridge sites.

A preliminary design for an infrared dryer was begun and almost immediately problems began to appear. The standard full-size heater is 8 feet (2.4 m) wide by 4 feet (1.2 m) long. To cover a 16-foot (4.9-m) wide section of bridge, two units, side by side are required. If only a 4-foot (2.4-m) long heat unit is used, the rate of travel over a bridge would be approximately 1 foot (305 mm) per hour; thus, a 100-foot (30.5-m) long bridge would require 200 hours to dry if it were two lanes wide. With the addition of multiple heat units the drying time could be reduced but the cost of the dryer was increased. A preliminary design was made for a unit consisting of six heaters, 16 feet (4.9 m) wide by 12 feet (3.6 m) long. In this design the front row of heaters would heat the concrete to 300°F (149°C) with 1-hour exposure while the second and third row would maintain that temperature for the required 2 hours. The rate of travel of this system was computed as 4 feet (1.2 m) per hour or 50 hours to heat and dry a 100-foot (30.5-m) long two-lane bridge. The estimated cost of the dryer was \$35,000 to \$40,000. This cost, while perhaps feasible for a contractor involved with drying and treating a number of bridge decks, did not appear economical for the one-time use in treating a single bridge deck as specified in our research agreement. An additional drawback to the infrared dryer is the fact that as a source for polymerization heat it would be quite hazardous due to the inflammability of the monomer.

Due to these problems with the infrared heat source, attention was returned to the gas-fired space heater. These heaters are portable, can be obtained commercially in various sizes, and with the use of suitable hot air heating ducts can be arranged in a drying system such that the open flame of the burner is far removed from monomer or monomer vapors.

To investigate the requirements of hot air drying, a series of tests was performed in forced draft ovens. This drying technique

was tested and evaluated in the laboratory and subsequently in the field.

In the laboratory tests, 2-foot (610-mm) by 2-foot (610-mm) by 4-inch (100-mm) thick concrete slabs were taken directly from a 73°F (23°C) 100 percent relative humidity curing room after they reached 28 days' age and subjected to forced air drying in a 4-foot (1.22-m) by 4-foot (1.22-m) by 7-foot (2.1-m) oven. Before being placed in the oven the sides and bottoms of the slabs were insulated with 3/8-inch (9.5-mm) thick synthetic foam rubber. The slabs were placed on a 3/4-inch (19-mm) thick plywood panel on the oven floor to reduce water loss from the bottoms of the slabs.

Previous experience during the PIC development program indicated that overnight drying at 300°F (149°C) was sufficient to permit full polymer impregnation under the proper conditions. In this series of tests, however, it was felt that lower drying temperatures could be used to remove the moisture from the top 1 to 2 inches (25 to 51 mm) of the concrete and the tests were performed at oven temperatures of 225° to 250°F (107° to 121°C). Initially, the test slabs were removed from the drying oven at 24-hour intervals and weighed to determine their water loss. These data, however, could not be correlated with polymer penetration and this step of the drying tests was discontinued. At the conclusion of the drying test the respective slabs were removed from the oven and allowed to cool to ambient temperatures. They were then impregnated with a monomer system composed of 95 percent MMA - 5 percent TMPTMA catalyzed with 1 percent AMVN using a soaking technique, which subsequently became the recommended field technique. Polymerization was accomplished by wrapping the impregnated slabs in polyethylene and returning them to an oven at 158°F (70°C).

Upon completion of the polymerization cycle the slabs were broken and inspected for polymer penetration. A summary of the results of these tests is presented in table 8. These data show that polymer penetrations of 3/4 to 1-3/4 inches (19 to 44 mm) were achieved with drying cycles of 24 to 60 hours at 250°F (121°C). It was not known, however, if the drying efficiency of the laboratory oven could be achieved in a large-scale hot air dryer for field applications and a conservative drying cycle of 60 hours at 250°F (121°C) concrete surface temperature was selected for use in the field demonstration.

In order to accomplish this drying cycle in the field, a hot air system was designed which included a 2-foot (610-mm) high aluminum-framed enclosure to retain the heat supplied by two or more gas-fired space heaters. Sheet metal ducts containing standard heat registers were used to distribute the hot air over the concrete surface within the enclosure. Temperature control within the

Table 8

SUMMARY OF HOT AIR DRYING TESTS

Specimen <u>1</u> / size ft x ft x in.	Temperature °F	Time hours	Water loss %	Polymer depth in.
2 x 2 x 4	225	24 48	2.6 3.6	- 1-1/2
2 x 2 x 4	230	48	3.7	1
2 x 2 x 4	230	24 48	3.0 4.1	- 1-1/2
2 x 2 x 4	230	24 48 60	3.2 4.2 4.6	- - 1-7/8 - 1-1/2
2 x 2 x 4	250	24	-	1-1/4 - 1-1/2
2 x 2 x 4	250	24	-	3/4 - 1
2 x 2 x 4	250	24	-	3/4 - 1
2 x 2 x 4	250	36	-	1 - 1/4
2 x 2 x 4	250	36	-	1 - 1/4
2 x 2 x 4	250	60	-	1-3/4
2 x 2 x 4	250	60	-	1-3/4
2 x 2 x 4	250	60	-	1-3/4

Table 8 - Continued

Specimen <u>1</u> / size ft x ft x in.	Temperature °F	Time hours	Water loss %	Polymer depth in.
Tests with specimens stored for 3 months in ambient air conditions				
2 x 2 x 4	250	24	-	1 - 1-1/4
2 x 2 x 4	250	36	-	1-1/8 - 1-3/8
2 x 2 x 4	250	48	-	1-1/2 - 1-5/8
2 x 2 x 4	250	60	-	1-3/4 - 1-7/8

1/Tests were performed immediately after specimens were exposed to 28 days or longer fog curing at 100 percent relative humidity.

enclosure was achieved by a proportioning thermistor controller which cycled the heaters to maintain the selected temperature set point. This drying system, when used with reliable heaters and control equipment, performed very well in two small-scale field tests and in the full-bridge demonstration treatment. A more detailed discussion of the equipment, heater capacity, and temperature maintenance is included in section 5, Field Tests.

3.3 Impregnation Studies

In the beginning of this research program it was thought that the impregnation step of the surface treatment process would be one of the most difficult to accomplish. Previous work performed during the PIC program indicated that first, it was difficult to impregnate to some specified depth such as 1 inch (25 mm), and second, once monomer penetration had occurred to the desired depth, it was difficult to maintain that depth during the polymerization process. This anticipated difficulty was encountered in the early impregnation studies. Numerous specimens were treated with several impregnation techniques with what appeared to be very discouraging results in obtaining a uniform and sufficiently deep penetration depth. As the overall process technology advanced, however, the difficulties previously believed to be caused by poor impregnation were found to be problems resulting from the other steps in the process. Ultimately, it was found that when the selected monomer system was applied to properly dried concrete surfaces, protected from evaporation, and subsequently polymerized promptly, the impregnation step was the simplest and most foolproof of the process.

Four impregnation techniques were tested. The results of the tests are shown in table 9. The first technique, ponding, was evaluated by calking a stainless steel ring, 3 inches (76 mm) high, onto the top surface of concrete slabs and using this ring to form a reservoir for monomer as shown in figure 5. Various soak times were used for monomer to penetrate the concrete surface. From 2 to 4 hours were required for a monomer penetration of 1 inch (25 mm) or more into previously dried concrete. This technique was used to evaluate laboratory drying and polymerization techniques and worked quite well on the 2- to 4-foot (610-mm to 1.2-m) square concrete slabs used in these tests.

The second technique was a combination of vacuum with ponding. In this method a welded plate steel enclosure was placed on a neoprene gasket on the surface of the test slabs and held in place with clamps. Figure 6 shows the enclosure and gasket being placed on a test slab. Vacuum was applied to the enclosure at 23 inches (584 mm) Hg and maintained for 15, 20, or 30 minutes. At the completion of the vacuum cycle, monomer was introduced into the enclosure and the vacuum was then released. The ponded monomer was allowed to soak into the concrete for 45 to 105 minutes to complete the impregnation cycle. This technique increased

Table 9

IMPREGNATION TESTS ON CONCRETE SLABS

Test No.	Technique	Soaking time hours	Monomer % MMA - % TMPTMA - % Catalyst	Polymer depth in.
13	<u>1</u> / P	1-3/4	90 MMA - 10 TMPTMA - 1 AIBN	1-1/8-1-1/4
14	<u>1</u> / P	1-3/4	90 MMA - 10 TMPTMA - 1 AIBN	1-1/4
16	<u>1</u> / P	2	90 MMA - 10 TMPTMA - 1 AIBN	1-1/4
51	<u>1</u> / P	2	90 MMA - 10 TMPTMA - 1 AIBN	1-1/4
11	<u>1</u> / P	4	MMA - 1 AIBN	1-3/4
12	<u>1</u> / P	4	90 MMA - 10 TMPTMA - 1 AIBN	1-3/4
20	<u>2</u> / V-P	<u>3</u> / 1/4-3/4	90 MMA - 10 TMPTMA - 1 AIBN	1-1-1/4
19	<u>2</u> / V-P	<u>3</u> / 1/2-1-1/2	90 MMA - 10 TMPTMA - 1 AIBN	1-1/4-1-3/8
30	<u>2</u> / V-P	<u>3</u> / 1/3-1-3/4	90 MMA - 10 TMPTMA - 1 AIBN	1-1/4-1-3/8
32	<u>2</u> / V-P	<u>3</u> / 1/4-1-3/4	90 MMA - 10 TMPTMA - 1 AIBN	1-1/2-1-3/4
21	<u>4</u> / V-P5	1/2-3/4	90 MMA - 10 TMPTMA - 1 AIBN	1-1/2
33	<u>4</u> / V-P7	1/4-1-3/4	90 MMA - 10 TMPTMA - 1 AIBN	1-1/2
65	<u>5</u> / S1	12	90 MMA - 10 TMPTMA - 1 AIBN	1-1/4-1-1/2
68	<u>5</u> / S1	16	90 MMA - 10 TMPTMA - 1 AIBN	1-1/2
76	<u>6</u> / S2	18	90 MMA - 10 TMPTMA - 1 AIBN	1-1/4-1-1/2
78	<u>6</u> / S2	18	90 MMA - 10 TMPTMA - 1 AIBN	1-1/4-1-1/2
72	<u>6</u> / S2	19	90 MMA - 10 TMPTMA - 1 AIBN	1-3/8-1-1/2
84	<u>6</u> / S2	18	90 MMA - 10 TMPTMA - 1 AMVN	1-1/2
85	<u>6</u> / S2	18	90 MMA - 10 TMPTMA - 1 AMVN	1-1/4
112	<u>6</u> / S2	4	95 MMA - 5 TMPTMA - 1 AMVN	1-1-1/4
113	<u>6</u> / S2	8	95 MMA - 5 TMPTMA - 1 AMVN	1-3/8-1-1/2
114	<u>6</u> / S2	12	95 MMA - 5 TMPTMA - 1 AMVN	1-3/4
94	<u>6</u> / S2	18	95 MMA - 5 TMPTMA - 1 AMVN	1-3/4-2
103	<u>5</u> / S1	18	97 MMA - 3 TMPTMA - 1 AMVN	1
102	<u>7</u> / S3	18	97 MMA - 3 TMPTMA - 1 AMVN	2
106	<u>6</u> / S2	8	95 MMA - 5 TMPTMA - 1/2 AMVN	1-1/4-1-1/2
104	<u>6</u> / <u>8</u> / S2	18	95 MMA - 5 TMPTMA - 1 AMVN	1-1/4-1-1/2

1/ P = Ponding.

2/ V-P = Vacuum at 23 in. Hg. followed by ponding.

3/ Time at vacuum/time of ponding.

4/ V-P5 or 7 = Vacuum at 23 in. Hg. followed by ponding under 5 or 7 lb/in² pressure.

5/ S1 = Saturated sand technique with one monomer application.

6/ S2 = Saturated sand technique with two monomer applications.

7/ S3 = Saturated sand technique with three monomer applications.

8/ Slab impregnated and polymerized while on 10 percent slope.



Figure 5. - Ponding reservoir on test slab.

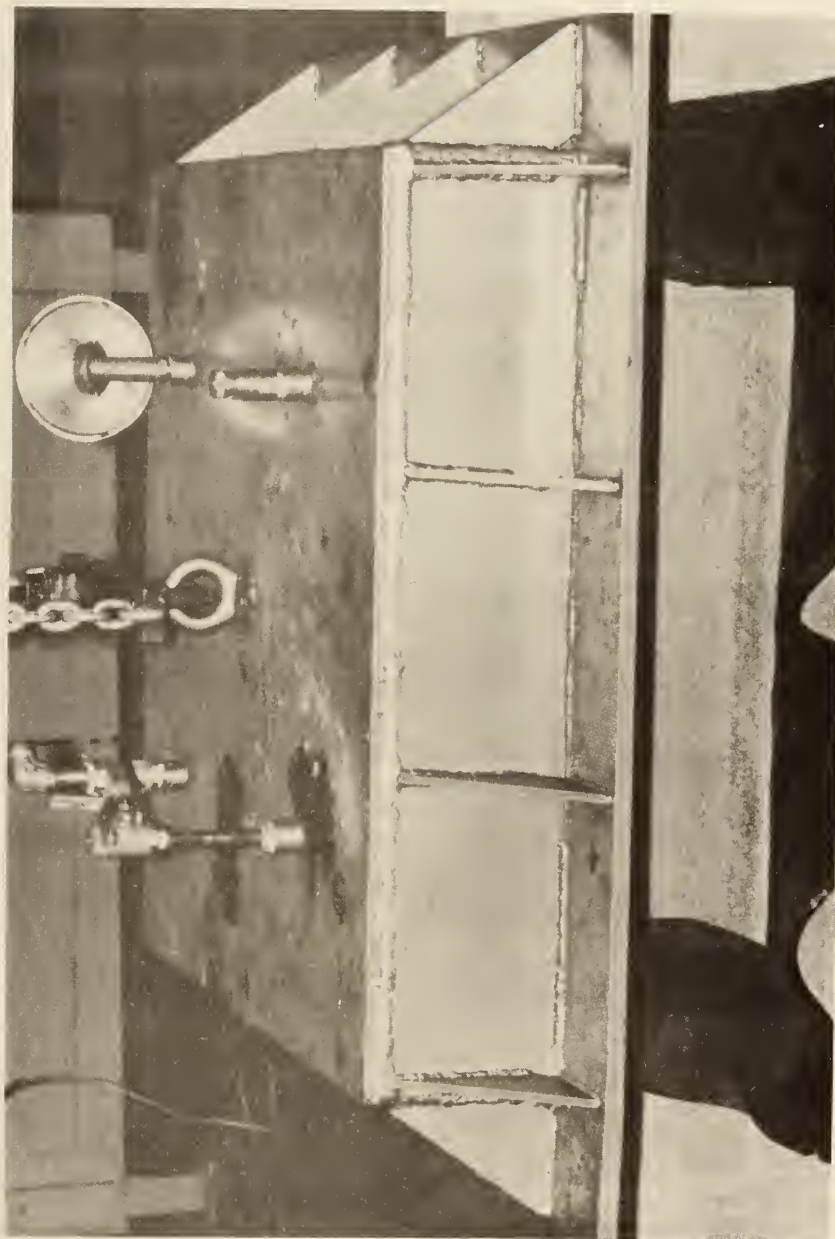


Figure 6. - Surface impregnator over concrete slab. Top contains fittings for vacuum, compressed air and monomer lines.

the rate of monomer penetration over that of ponding alone and achieved the same penetration depth in approximately one-half the time.

A shorter impregnation cycle was desirable and to accomplish this a third impregnation method, vacuum-ponding at pressure, was tested. These tests were performed using the same procedures as the vacuum-ponding tests except that during the monomer soaking cycle, compressed air at a pressure of 5 or 7 lb/in² (34.5 or 48.3 x 10³ N/m²) was applied to the enclosure. This, of course, required a tighter seal between the enclosure and the concrete slabs. In comparison with the ponding and vacuum-ponding methods, the pressure impregnation, at the relatively low pressures tested, did not produce sufficient benefit to justify contending with the problems of monomer leakage. If, however, penetration depths greater than 1 inch (25 mm) had been desired, the pressure impregnation technique may have been practical.

The fourth impregnation technique tested was the saturated sand method. With this method of impregnation a 1/4- to 3/8-inch (6.4- to 9.5-mm) thick layer of dry sand is spread over the dried concrete surface and fully saturated with monomer. The monomer is then allowed to soak into the concrete. The sand serves two basic purposes: it acts as a reservoir, holding the monomer on the concrete surface, and it reduces monomer evaporation during the soaking period. To further reduce evaporation, the monomer-saturated sand is covered with a polyethylene membrane. In the initial developmental tests of this method, only one monomer application was used. It was subsequently found that deeper polymer penetration resulted from making two or three monomer applications spaced approximately 2 to 4 hours apart. In these tests, AMVN catalyst was used with a 95-5 MMA-TMPTMA monomer system. Both 4- and 8-hour soak cycles resulted in adequate monomer penetration to achieve the desired polymer depths in the laboratory (tests No. 112, 113, 114, and 94). Under field conditions an 8-hour soak cycle may be needed to allow for the various unknown factors that might be present.

Several gradations of sand were used to retain the monomer on the concrete surface. Generally, sand size, within limits, had little effect upon monomer penetration; the limits being that particle sizes greater than a No. 16 sieve or smaller than a No. 100 sieve were usually undesirable. Particle sizes larger than No. 16 permitted the monomer to flow off the concrete surface while those smaller than No. 100 tended to hold the monomer in the sand rather than allowing it to penetrate the concrete.

The sand gradation that gave best results in the laboratory and subsequently worked very well in the field is as follows:

100 percent passing a No. 16 sieve with roughly equal weights retained on No. 30, No. 50, and No. 100 sieves. Ideally, not more than 2 percent of the sand particles should pass a No. 100 sieve.

It should be pointed out that in many cases washed pit-run sand suitable for high quality concrete will be adequate for use with this impregnation technique. Care should be taken, however, to assure that any sand used is uniformly blended and dry before monomer application.

A review of test results in table 9 shows that each of the four methods provided polymer penetrations that equaled or exceeded the desired 1 inch (25 mm). Of these methods, the saturated sand technique was ultimately chosen as the most suitable and practical field method. Each of the other methods had one common drawback, that being the problem of ponding monomer on sloped concrete surfaces. Instances of up to 5 percent bridge deck superelevation on curves are very common. In the case of a 32-foot (9.7-m) wide bridge deck, a superelevation of 5 percent gives the roadway surface a 19.2-inch (490-mm) elevation change across its width. A series of reservoirs would be required for ponding across such a surface. Providing leakproof seals for these reservoirs would be understandably troublesome. There would also be the additional problem of handling excess catalyzed monomer, left over at completion of the ponding cycle. The saturated sand impregnation technique utilizes only the monomer required for concrete penetration with allowances for evaporation. In laboratory tests on slabs at a 10 percent slope (test No. 104) and in a full-scale bridge treatment subsequently performed by FHWA on a bridge with 5 percent superelevation this technique gave uniform polymer penetrations of 1 to 1-1/2 inches (25 to 38 mm) (see Section 5.4, Additional Full-size Bridge Treatments).

During tests of impregnation techniques it was found that the monomer systems catalyzed with AIBN or AMVN were quite sensitive to solar radiation. Test slabs placed in sunlight during the impregnation cycle suffered monomer polymerization on their surface within 20 to 40 minutes. Such an occurrence eliminates the availability of monomer for soaking into concrete to the desired depth, causes a barrier to be formed on the surface of the concrete which precludes additional monomer applications at periodic intervals, and results in conversion of the monomer-saturated sand layer to a polymer mortar which may become tightly bonded to the underlying concrete and, therefore, may be difficult to remove. For this reason it is necessary to shade the concrete during impregnation or to perform the impregnation cycle between sunset and sunrise. It should be pointed out that although sunlight can cause polymerization of monomer on the surface of concrete, it is not sufficient to polymerize monomer impregnated into concrete to a depth of 1 inch (25 mm).

3.4 Polymerization Studies

Polymerization is a chemical reaction whereby large numbers of individual monomer molecules are linked together to form macromolecules of polymer. Basically there are two types of polymerization reaction, step polymerization and chain polymerization. These reactions yield condensation or addition polymers, respectively. Examples of condensation polymers include polyesters, polyurethane, and naturally occurring polymers such as protein, wool, and silk. Addition polymers include polyethylene, poly(vinyl chloride), polystyrene, and poly(methyl methacrylate). This study is concerned with the chain polymerization mechanism and the resulting addition polymers.

The chain polymerizations are normally dependent upon a catalyst which decomposes to produce an initiator with a reactive center. Through this reactive center successive linking of large numbers of monomer molecules (addition) occur in a chain reaction in combination with the propagation of the reactive center. This linking continues until the reactive center is destroyed by one of several possible terminal reactions.

Translating this explanation of the polymerization process to practical application, the requirements for polymerization thus become a polymerizable monomer, a polymerization catalyst, and a technique of decomposing the catalyst to generate the necessary reactive centers in sufficient quantity to permit linking of all the monomer molecules. ^{1/} These requirements are interdependent in that a change of monomer and/or catalyst will generally require a change or modification of polymerization technique.

In this study two basic techniques were investigated. The first involved the use of a chemical promoter which when added to catalyzed monomer caused the catalyst to decompose at ambient temperatures. In this report this technique is referred to as promoter-catalyst polymerization. The second technique involved the addition of heat, which due to temperature sensitivity of the catalyst, caused its decomposition. This technique is known as thermal-catalytic polymerization. Of the two techniques, the promoter-catalyst process would be the most desirable for bridge deck applications if it were controllable to the degree required. Unfortunately, the surface impregnation process contains inherent limitations within which it is difficult to utilize this polymerization technique.

^{1/} For practical purposes it can be said that all molecules are linked. In reality something less than 100 percent monomer conversion is usually achieved.

Polymerization of acrylic monomers is an exothermic reaction. The heat generated by the reaction causes a temperature increase in the polymerizing monomer which in turn increases the rate of polymerization. In the polymerization of bulk quantities of monomer using the promoter-catalyst technique low promoter-catalyst ratios can be used to initiate the reaction which in turn generates sufficient heat to further decompose the catalyst and results in complete polymerization. With low promoter concentrations the time period between addition of promoter to the catalyzed monomer and the noticeable increase in monomer viscosity that occurs with onset of polymerization can be controlled to the point that sufficient time is available for the monomer to impregnate concrete to the desired depth. Once this impregnation occurs, however, the polymerization conditions are vastly changed. The monomer is then an integral part of a vast heat sink which absorbs the exotherm heat and prevents any significant increase in monomer temperature. The catalyst therefore does not generate sufficient reactive centers and polymerization does not occur or does not go to completion. A solution to this problem is to increase the concentration of promoter in the monomer. More reactive centers are thus generated from the catalyst within a shorter period of time and the monomer system becomes independent of polymerization heat for a complete reaction. Unfortunately, when sufficient promoter is added to accomplish this, the time between addition of promoter and the viscosity increase associated with polymerization is reduced to the point that impregnation to the desired depth does not occur.

Table 6 lists the results of attempts at polymerization with promoter-catalyst systems. These systems did not yield the degree of control required in the surface impregnation process. However, with the numerous promoters and catalysts commercially available for polymerization of acrylic monomers it is possible that with additional study a system which meets the needs of this process may be found.

Thermal-catalytic polymerization techniques have been utilized in concrete-polymer materials research since the beginning of the program at the Bureau and through a series of screening tests one catalyst, α -t-butylazo isobutyronitrile, was selected as the standard for use in polymerizing precast polymer impregnated concrete. This catalyst, as previously mentioned, requires relatively high temperature to initiate polymerization. Another azo catalyst, AIBN, has a lower decomposition temperature and was initially selected for polymerization of surface impregnated concrete. The problem then became one of selecting a practical method of safely heating an impregnated concrete bridge deck to the required AIBN decomposition temperature while preventing excessive monomer loss due to evaporation.

This appeared relatively easy to accomplish in the laboratory with small concrete specimens. Impregnated concrete slabs were wrapped in aluminum foil or polyethylene plastic and placed in forced draft electrical ovens for polymerization. With 1/2 to 1 percent AIBN as catalyst in either 90-10 or 95-5 MMA-TMPTMA monomer systems, polymerization was accomplished at 176°F (80°C). With this technique excessive monomer evaporated from the concrete and left a 1/4- to 3/4-inch (6.4- to 19.0-mm) deep zone at the surface of the concrete that contained little or no polymer. To prevent this, an excess of monomer was added to the concrete surface just prior to polymerization heat application. This was only partially successful because the impregnated concrete surface generally did not retain sufficient excess monomer to meet the evaporation requirements during polymerization. In an attempt to hold the excess monomer on the surface several layers of rags were placed on the impregnated concrete and saturated with monomer. The entire slab was wrapped in polyethylene and placed in an oven at 176°F (80°C) for overnight polymerization. The resulting polymer penetration was 1-1/4 to 1-1/2 inches (32 to 38 mm) deep and uniform to the surface of the concrete. Additional slabs were polymerized in this manner, each obtaining a uniform polymer penetration. During these tests it was found that it was unnecessary to wrap the entire slab in polyethylene and that only the top surface and vertical sides needed to be covered with the protective film. Although not too practical for field applications, this polymerization technique was adopted as a laboratory standard and used to polymerize numerous test slabs during the development of the drying and impregnation techniques. An oven temperature of 176°F (80°C) and an overnight heat cycle were convenient and reliable to use with this polymerization technique.

Investigations of other thermal-catalytic polymerization techniques were continued to develop a practical field procedure. Among these were the use of hot water and steam to heat the impregnated concrete. The use of hot water as a heat source for polymerization had been investigated and adopted in the PIC process both at the Bureau and at Brookhaven National Laboratory and appeared to be potentially useful for the surface impregnation process. Two basic types of hot water polymerization techniques were evaluated: hot water in direct contact with the impregnated concrete and hot water separated from the concrete surface by polyethylene film. The first method, hot water in direct contact with the concrete, did not appear satisfactory as polymer was found in the water. The second method appeared more promising and was investigated in more detail. A bottomless wood reservoir was constructed and lined with the polyethylene film which provided efficient heat transfer to the concrete. Figure 7 shows the reservoir being used on a test slab.



Figure 7. - Hot water polymerization reservoir.

The test results (tests No. 29, 38, 39, and 40 of table 10) showed that water at 180°F (82°C), ponded to a depth of 2-3/4 inches (69.8 mm) over the concrete, did not provide the required heat, and that additional heating was necessary. Additional heat was provided by a continuous flow of hot water at the selected polymerization temperature. Tests No. 42 through 58 of table 10 show the results of this technique. Generally, it was found that 5 to 7 hours exposure to 160°F (71°C) water temperature was required to accomplish polymerization. Slightly better polymer penetrations were achieved with a 2 percent AIBN concentration than with the 1 percent concentration. The use of the catalyst-promoter system in test No. 54 did not improve the polymer penetration or appearance. Generally, all these tests showed the irregular, splotchy zone associated with excessive monomer evaporation prior to polymerization. In order to reduce the evaporation, excess monomer and additional heat was applied to the concrete. In test No. 60 the water temperature in the polymerization reservoir was raised to 200°F (93°C) and maintained at that temperature for 5 hours. This was not effective and in test No. 64 additional heat was supplied to the bottom of the impregnated concrete slab with steam while the water reservoir temperature was maintained at 160°F (74°C). Monomer-saturated rags were placed on the concrete surface beneath the water reservoir. The resulting polymer penetration was uniform and to the desired depth. Similar results were obtained by replacing the rags with monomer-soaked sand (test No. 65). Tests No. 66 through 74 were performed as checks to confirm the results of the previous tests.

These tests indicated that monomer evaporation from the concrete during the polymerization cycle was the major cause of the less than satisfactory polymer penetrations. When this evaporation was reduced either through providing excess monomer or additional heat for a more rapid polymerization, the resulting polymer penetrations were to the desired depth and density.

In test No. 76 an additional layer of dry sand was placed over the monomer-saturated sand (the sand layers were separated by a polyethylene sheet) and used simply as weight to further seal the impregnated surface and prevent excessive monomer evaporation (the same effect possibly could be accomplished by increasing the water depth in the reservoir). A uniform polymer penetration of 1-1/4 to 1-1/2 inches (32 to 38 mm) was achieved. These results were subsequently repeated on four additional test slabs (No. 78 through 81).

AMVN polymerization catalyst, which appeared potentially very useful for polymerization of the surface impregnated concrete, was used in tests No. 84 and 85. Polymerization screening tests with AMVN and the 90-10 MMA-TMPTMA monomer system indicated that polymerization

Table 10

HOT WATER POLYMERIZATION TESTS OF
IMPREGNATED CONCRETE SLABS

Test No.	Monomer/technique	Results
29	90-10-1 AIBN - 4-gallon 150°F water placed on surface separated by polyethylene. Then placed in 175°F oven overnight	No polymerization. Water cool to touch due to evaporation
38	90-10-1 AIBN - 175°F water placed on concrete surface and covered to prevent evaporative cooling. Placed in 170°F oven overnight	No polymerization in slab. Polymer-water slurry on concrete surface
39	90-10-1 AIBN - 180°F water in direct contact with concrete. No additional heating of water	Water-polymer slurry on surface. No polymerization in concrete
40	90-10-1 AIBN - 180°F water with polyethylene between water and concrete. No additional heat added to water	Polymer skin on concrete surface. No polymer in concrete
42	90-10-2 AIBN - Polyethylene water reservoir with 160°F water temperature maintained for 1 hour then allowed to cool to ambient	1/4- to 3/8-inch polymer penetration
43	90-10-2 AIBN - Same as 42 except 175°F water temperature maintained for 3 hours then allowed to cool	5/8-inch polymer penetration
44	90-10-2 AIBN - Same as 42 except 175° to 180°F water temperature maintained for 5 hours then allowed to cool	7/8- to 1-inch polymer penetration
49	90-10-1 AIBN - Polyethylene water reservoir placed over concrete. 175°F water temperature maintained for 7-1/2 hours	3/4- to 7/8-inch polymer penetration with discontinuous-splotchy appearance

Table 10 - Continued

Test No.	Monomer/technique	Results
52	90-10-1 AIBN - 7-1/2 hours with polyethylene water reservoir at 160°F. 1/4-inch monomer saturated sand on concrete surface	3/4-inch polymer penetration, dense, uniform
54	90-10-2 BP and 0.15 percent DMA. 5 hours with polyethylene water reservoir at 160°F	1-inch polymer penetration with discontinuous-splotchy appearance
58	90-10-2 AIBN - 5 hours with polyethylene water reservoir at 160°F	1-inch polymer penetration with splotchy appearance
60	90-10-1 AIBN - 5 hours with polyethylene water reservoir at 200°F (boiling). 1/4-inch monomer saturated sand on concrete surface	1-inch polymer penetration with irregular, splotchy appearance
64	90-10-1 AIBN - 5 hours with polyethylene water reservoir at 160°F. Monomer saturated rags on concrete surface. 185°F steam directed onto bottom of slab	1-1/4- to 1-1/2-inch uniform polymer penetration
65	90-10-1 AIBN - 5 hours with polyethylene water reservoir at 160°F. 1/4-inch monomer saturated sand on concrete surface. 185°F steam directed onto bottom of slab	1-1/4- to 1-1/2-inch uniform polymer penetration
66	90-10-1 AIBN - 5 hours with 160°F polyethylene water reservoir. 1/4-inch monomer saturated sand on concrete surface	Average depth of penetration 1/2 inch with one area extending to 1-1/2-inch

Table 10 - Continued

Test No.	Monomer/technique	Results
67	Repeat of 66 for use at FHWA Fairbank Laboratory chloride intrusion test	1/2- to 3/4-inch irregular, splotchy polymer penetration
68	Repeat of hot water-steam as in No. 65	1-1/2-inch uniform polymer penetration
72	90-10-1 AIBN - 5-1/2 hours with 160°F polyethylene water reservoir. 1/4-inch sand and monomer saturated rags on concrete	1-3/8- to 1-1/2-inch uniform, dense polymer penetration
74	Same as No. 72 but without sand - rags only	1- to 1-1/2-inch dense polymer penetration
76	90-10-1 AIBN - 4 hours with 165°F polyethylene water reservoir. 1/4- to 3/8-inch monomer saturated sand on concrete surface. Additional 3/8-inch layer of dry sand placed over monomer saturated sand - separated by polyethylene sheet to act as weight	1-1/4- to 1-1/2-inch dense polymer penetration
78, 79, 80, 81	Same as No. 76 to confirm results	1-1/4- to 1-1/2-inch dense polymer
84	90-10-1 AMVN - 5-1/2 hours with 130° to 140°F polyethylene water reservoir directly on concrete surface. Excess monomer added to concrete surface prior to polymerization	1-1/2-inch polymer penetration. Uniform, dense
85	Same as No. 84 except no excess monomer added to surface of concrete	1-1/4-inch polymer penetration. Uniform to surface

temperatures of 130° to 140°F (55° to 60°C) resulted in polymerization at a rate faster than that achieved with AIBN at 160° to 170°F (71° to 77°C). In test No. 84 the test slab was polymerized using a 130° to 140°F (55° to 60°C) hot water reservoir placed directly on the impregnated concrete surface. An excess of monomer was added to the concrete just prior to polymerization. After 5-1/2 hours exposure to the heat of the water the reservoir was removed and the slab broken to determine polymer depth. A uniform, dense, polymer penetration of 1-1/4 to 1-1/2 inches (32 to 38 mm) was visually evident in the concrete. This polymerization system was repeated in test No. 85 but without the addition of excess monomer prior to polymerization. The resulting polymer penetrations exceeded the desired 1-inch (25-mm) depth and in no instance was excessive evaporative loss of monomer apparent.

This polymerization technique with AMVN catalyst appeared feasible for field application. The initial design of this field polymerization system was begun but the same problems encountered on sloped surfaces with the ponding impregnation technique were evident. In addition, the problems of maintaining the desired water temperature for 4 to 6 hours and of recirculating the water for heating (the water would probably be contaminated by varying amounts of monomer and/or polymer due to leaks in the reservoir lining) became apparent. Faced with these considerations somewhat more exotic concepts evolved such as semirigid hot water bladders. The costs of such systems were excessive for a one-time field demonstration, however.

Two attempts were made at polymerizing surface impregnated concrete with live steam. In each of these tests excessive evaporation of monomer occurred due to a faulty temperature control system even though the impregnated concrete was wrapped in polyethylene film and contained an excess of monomer on its surface. A third attempt at steam polymerization involved the use of steam to heat impregnated concrete from the bottom surface of the test slab. Somewhat surprisingly, this technique gave very good results but was not investigated further because it was not considered practical for field applications with the steam generating equipment available at that time.

During the studies of these polymerization techniques the investigations of drying and impregnation techniques were also being performed. In these investigations polymerization was generally accomplished in the forced draft ovens as previously explained and substantial experience with hot air polymerization was gained. With the selection of forced hot air as a field drying technique, the advantages of utilizing the same technique for polymerization of the impregnated concrete became obvious. The equipment used to heat concrete to the desired drying temperature could easily meet the lower temperature requirements of polymerization. With the

use of AMVN catalyst, polymerization occurred at a faster rate and at a lower temperature than with the AIBN catalyst and the problem of monomer evaporation was accordingly not as severe. A series of tests was performed to determine optimum hot air polymerization temperatures and durations for slabs impregnated with the AMVN catalyzed monomer system. In these tests, the saturated sand impregnation technique was used and the monomer dampened sand was left on the concrete surface and covered with polyethylene film during polymerization. At the time these tests were performed it was not known what technique would be used to monitor and control polymerization temperatures, i.e., temperature control based on concrete surface temperature or measurements of air temperature immediately above the concrete. For this reason both types of temperature measurement were evaluated. Using the concrete surface temperature it was found that concrete impregnated with the 90-10 MMA-TMPTMA monomer systems catalyzed with 1 percent AMVN could be polymerized by obtaining a 130° to 140°F (55° to 60°C) surface temperature and maintaining that temperature for a minimum of 4 hours. The results of these tests are shown in table 11. The durations listed are the times the concrete surfaces were at the indicated temperatures and do not include the heat-up times required to reach the listed surface temperatures.

Using the air temperature approach, impregnated concrete slabs were placed in a preheated oven and exposed to the test temperatures for varying times. The oven used in these tests was 4 feet (1.2 m) wide by 4 feet (1.2 m) high by 7 feet (2.1 m) long and contained an air circulation fan approximately 3 feet (910 mm) in diameter. Temperature uniformly within this oven was very good. With this approach it was found that a minimum of 6 hours exposure to 158°F (70°C) was required to obtain the desired polymerization. These test results are shown in table 12.

With respect to the impregnation and polymerization of MMA in concrete, there is evidence that unpolymerized MMA in the presence of water will react with calcium hydroxide of portland cement to form the salt, calcium methacrylate. A preliminary study of this reaction was performed at the Bureau in 1973. 2/ In this study it was found that the calcium methacrylate salts were highly soluble in water and in the quantities likely to be formed in time periods normally experienced between impregnation and polymerization did not appear harmful in any way to the concrete or the enhanced durability of the polymer impregnated concrete. After polymerization there is no further reaction nor is there any evidence of reaction between poly(methylmethacrylate) and concrete. Less well defined, however, is the possibility of harm to concrete through long-term exposure to MMA monomer. This is the situation that could exist due to partial or incomplete

Table 11

POLYMERIZATION TESTS BASED ON CONCRETE
SURFACE TEMPERATURE
90-10 MMA-TMPTMA with 1 Percent AMVN

Test No.	Concrete surface temperature ° F	Duration hours	Polymer depth in.
88	113 to 122	18	5/8-3/4
94	122 to 131	5-1/2	1-3/4
90	131 to 140	3	1-1/4
98	131 to 140	5-1/2	1-1/2- 1-3/4

Table 12
POLYMERIZATION TESTS BASED ON
OVEN TEMPERATURE

Test No.	Monomer system <u>1/</u> ,	Oven temperature °F	Duration hours	Polymer depth in.
96	90-10-1	158	18	1-1/2
97	90-10-1	158	5-1/2	1-1/4- 1-1/2
106	95-5-1/2	158	18	1-1/4- 1-1/2
115	95-5-1	140	2	0
116	95-5-1	140	4	0
117	95-5-1	140	6	1-1/2 with 1/2 inch evapora- tive loss
118	95-5-1	158	6	1-3/8- 1-1/2
119	95-5-1	158	8	1-1/2

1/ Monomer system listed as % MMA - % TMPTMA - % AMVN.

polymerization of impregnated concrete. In the absence of a definitive study of the effects of long-term monomer-concrete exposure a conservative approach to polymerization temperatures and times was justified to ensure that the monomer was converted to unreactive polymer.

With this approach and the results of the hot air polymerization tests, the technique of temperature control based on concrete surface temperature was selected to provide the least possibility of error in the field trials and the full scale demonstration test. Temperature uniformity and control would not be as precise in the field as in the laboratory and a minimum polymerization cycle of 6 hours duration at a minimum surface temperature of 160°F (71°C) was selected to provide a margin of safety against incomplete polymerization with either the 90-10 or the 95-5 MMA-TMPTMA monomer system containing 1/2 to 1 percent AMVN catalyst.

4. DURABILITY TESTS

Various tests of the durability of polymer impregnated concrete have been underway since the beginning of the concrete polymer materials program in 1966. The results of the tests have been reported in a series of five publications prepared jointly by the Bureau and Brookhaven National Laboratory. 3/ As a result of this work, most of the durability properties of PIC were well defined at the time the bridge deck surface impregnation program was begun and there was little need to repeat the previous tests during this program.

There were, however, three areas of durability that had not been tested and which were of concern in bridge deck applications. The first was chloride intrusion into impregnated concrete which was exposed to repeated applications of deicing salts. The second was the possibility that the impregnated zone might delaminate, crack, or separate from the unimpregnated zone during exposure to freezing and thawing; and the third was the skid and abrasion resistance properties of surface impregnated concrete.

4.1 Chloride Intrusion Test

Chloride intrusion tests were performed on surface impregnated concrete by the Federal Highway Administration's Fairbank Highway Research Station Laboratories. The concrete test specimens were cast and surface impregnated by the Bureau and sent to the Fairbank Laboratory for testing and evaluation. FHWA performed the tests and supplied the data included herein.

The test slabs were 4 feet (1.2 m) by 2 feet (610 mm) by 4 inches (102 mm) thick and contained two-way No. 4 reinforcing steel on 12-inch (305-mm) centers located 1 inch (25 mm) below the concrete surface.

Surface impregnation was performed with the MMA-TMPTMA monomer system using the saturated sand impregnation technique and thermal-catalytic polymerization at 158°F (70°C). Resulting polymer penetrations were 1-1/4 to 1-1/2 inches (32 to 38 mm) deep.

The impregnated slabs were tested as follows:

The slabs were located in an out-of-doors test yard and a butyl-rubber calking compound dam approximately 1/4 inch (6.4 mm) high was placed around the top surface of each slab. They were then subjected to daily ponding with a 3 percent sodium chloride solution to a depth of 1/16 inch (1.6 mm). This procedure resulted in a daily wet-dry surface during much of the

year. Freezing temperatures were also encountered during the winter months. Samples of the concrete were periodically taken by coring. In order to separate and evaluate various depths of concrete, the cores were then sliced without the aid of water, pulverized, and analyzed for chloride content utilizing the procedure described by Berman. ^{4/} These data for conventional and surface impregnated test specimens are shown in table 13.

In their studies ^{5/}, FHWA has determined that the chloride content corrosion threshold for portland cement concrete with a cement factor of 7.0 (658 lb cement per yd³) is approximately 1.3 pounds Cl per cubic yard of concrete (20.82 kg/m³). FHWA has determined that in some instances only about seven daily salt applications were necessary to reach this threshold at the 1-inch (25-mm) depth in conventional bridge deck concrete. After some 600 salt applications the chloride content at the 1-inch (25-mm) depth in the surface impregnated test slabs was less than 0.3 lb/ft³ (4.80 kg/m³).

Figure 8 is a plot of chloride content versus depth for surface impregnated concrete and for two conventional concretes. For this figure the chloride values of impregnated specimens No. 78 and 79 of table 13 were averaged at the respective depths for the impregnated concrete curve. After 600 salt applications, chloride contents greater than the corrosion threshold had penetrated past the 2-inch (51-mm) depth in conventional concrete, but less than the 1/2-inch (13-mm) depth in the impregnated concrete.

4.2 Freeze-thaw Delamination Tests

The concrete slabs used in this test were 2 feet (610 mm) wide by 6 feet (1.8 m) long by 7 inches (180 mm) thick. Each slab contained a two-way grid of deformed No. 4 reinforcing steel located on 1-foot (300-mm) centers and 2 inches (50 mm) below the top surface of the concrete. A stainless steel reservoir was calked onto the top surface of the concrete to retain the water used for the freeze-thaw test as shown in figure 9.

Weekly freezing and thawing cycles were performed as follows:

On Monday morning the slabs were moved into a -10°F (-23°C) freezer room and the water required to obtain a 1/4-inch (6.4-mm) depth added to the reservoir, as shown in figure 10. The slabs were left in the freezer room for 24 hours. On Tuesday morning the slabs were removed from the freezer and 1.3 pounds (590 gm) of sodium chloride salt were added to the surface of the ice, figure 11. After the ice began to thaw, flexible drain tubes in the reservoir were opened and the slabs left to thaw, drain, and dry for the remainder of the week.

Table 13

CHLORIDE CONTENT OF TEST SLABS A/

Slab No.	Sample depth inches	Chloride content - lb/yd ³			
		204 daily salt applications	330 daily salt applications	375 daily salt applications	600 daily salt applications
Control (W/C = 0.40, CF = 7.0)	0.25	-	3/ 9.2	-	-
	1.0	-	<u>10/ 0.7</u>	-	-
	2.0	-	<u>10/ 0.1</u>	-	-
	3.0	-	<u>10/ 0.1</u>	-	-
Control (W/C = 0.50, CF = 7.0)	0.25	-	3/ 12.7	-	3/ 33.0
	1.0	-	<u>10/ 3.6</u>	-	<u>3/ 8.6</u>
	2.0	-	<u>10/ 1.0</u>	-	<u>3/ 1.6</u>
	3.0	-	<u>10/ 0.1</u>	-	<u>3/ 1.0</u>
Surface Impregnated to 1-1/4 to 1-1/2 Inches					
78 (W/C = 0.45, CF = 6.5)	0.28	1/ 1.3	-	-	3/ 3.5
	0.75	<u>1/ 0.1</u>	-	-	<u>3/ 0.3</u>
	1.25	<u>1/ 0.1</u>	-	-	<u>3/ 0.3</u>
	1.75	<u>1/ 0.1</u>	-	-	<u>3/ 0.2</u>
79 (W/C = 0.45, CF = 6.5)	0.28	1/ 0.7	-	-	2/ 5.1
	0.75	<u>1/ 0.6</u>	-	-	<u>2/ 0.5</u>
	1.25	<u>1/ 0.0</u>	-	-	<u>2/ 0.3</u>
	1.75	<u>1/ 0.2</u>	-	-	-
80 (W/C = 0.45, CF = 6.5)	0.28	1/ 2.7	-	-	-
	0.75	<u>1/ 0.2</u>	-	-	-
	1.25	<u>1/ 0.2</u>	-	-	-
	1.75	<u>1/ 0.3</u>	-	-	-

Table 13 - Continued

Slab No.	Sample depth inches	Chloride content - lb/yd ³			
		204 daily salt applications	330 daily salt applications	375 daily salt applications	600 daily salt applications
81 (W/C = 0.45, CF = 6.5)	0.28	1/ 0.6	-	-	-
	0.78	<u>1/</u> 0.3	-	-	-
	1.25	<u>1/</u> 0.3	-	-	-
	1.75	<u>1/</u> 0.1	-	-	-
98 (W/C = 0.45, CF = 6.5)	0.28	-	-	2/ 3.3	-
	0.78	-	-	<u>2/</u> 0.3	-
	1.25	-	-	<u>2/</u> 0.3	-
	1.75	-	-	<u>2/</u> 0.4	-

A/ = Data supplied by FHWA Fairbank Laboratory.

CF = Cement factor - bags per cubic yard.

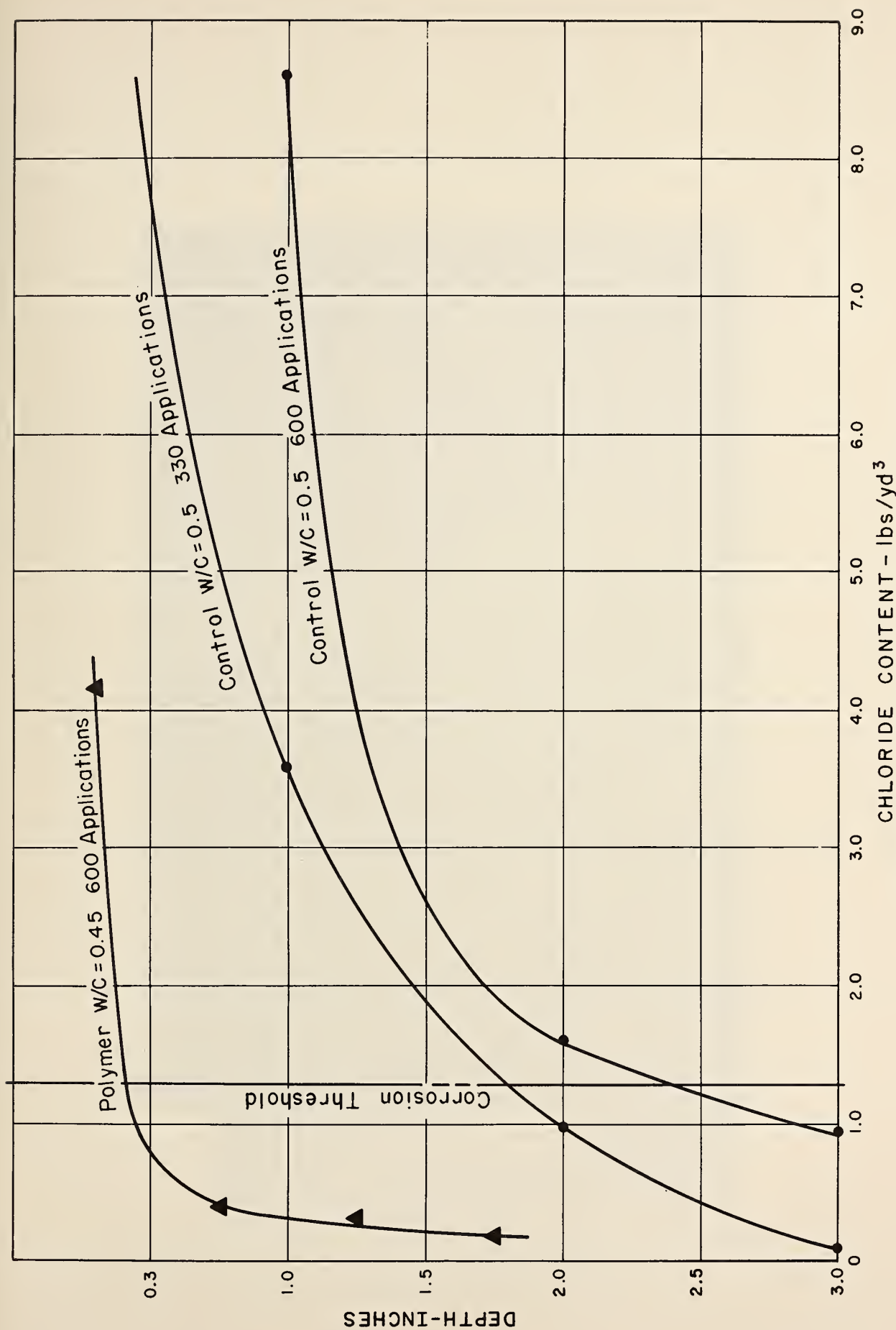
W/C = Water/cement ratio.

1/ = Single sample.

2/ = Average of two samples.

3/ = Average of three samples.

10/ = Average of 10 samples.



CHLORIDE CONTENT OF TEST SLABS

FIGURE 8



Figure 9. - Water reservoir on freeze-thaw test slab.
This reservoir was calked onto the concrete
to retain the water during freezing.

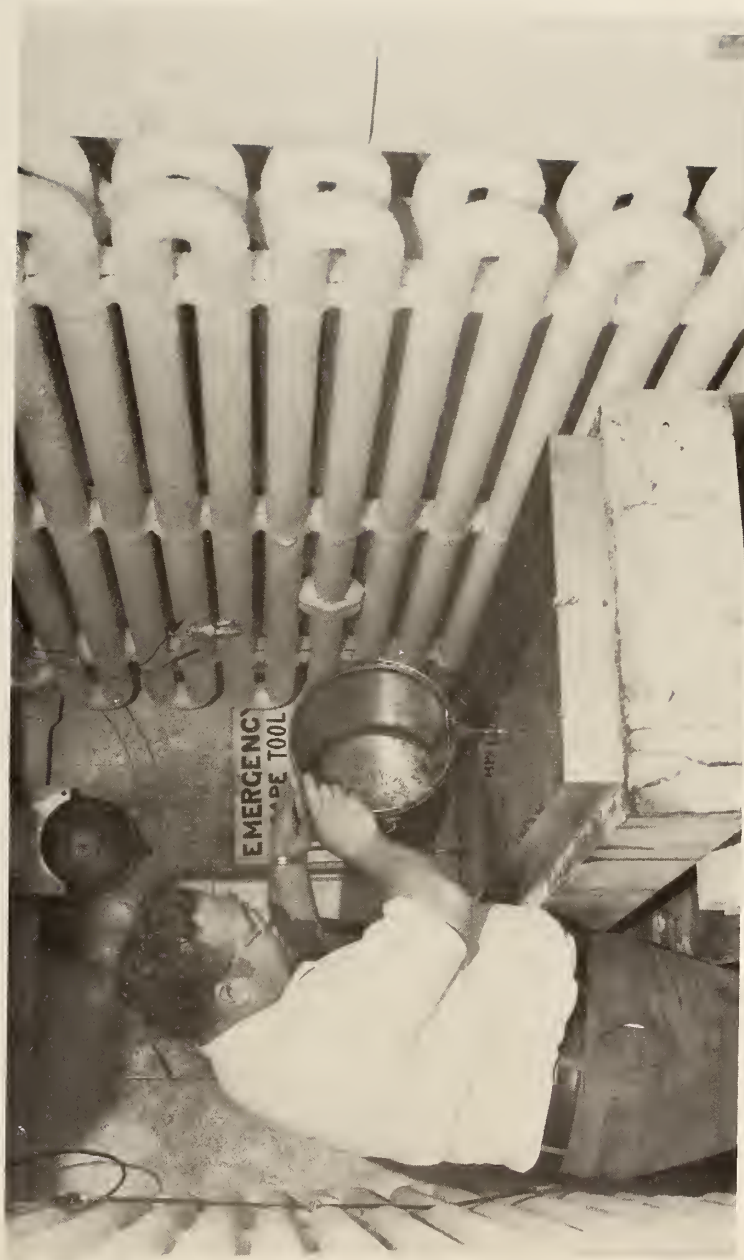


Figure 10. - Adding water to the freeze-thaw slab.



Figure 11. - Placing deicing salt on frozen surface.

It was originally planned to test a single impregnated slab in this manner. This slab was cast and dried at 350°F (177°C) at 14 days age. Impregnation was accomplished with the 90-10 MMA-TMPTMA monomer system containing 1 percent AIBN using the saturated sand technique. Polymerization was accomplished with 160°F (71°C) water.

Shortly after this slab was placed in the freeze-thaw test it was noticed that cracks were present directly over each transverse reinforcing bar. These cracks apparently had formed during the drying or polymerization cycle as a result of shrinkage of the relatively young concrete and high drying temperatures.

Two additional slabs were cast and cured in a 100 percent relative humidity environment for 28 days. One of these slabs was impregnated, the other remained untreated to serve as a control. The treated slab was oven-dried using a 30°F (17°C) per hour heat up rate to 300°F (149°C) followed by 13 hours drying at 300°F (149°C). Impregnation was accomplished with the saturated sand technique using the 95-5 MMA-TMPTMA monomer system and 1 percent AMVN. Oven polymerization at 140°F (60°C) completed the treatment and resulted in a polymer penetration of 1-1/4 to 1-1/2 inches (32 to 38 mm).

Freeze-thaw tests on the treated and control slabs were begun in June 1974 and continued until November 1975, for a total of 69 cycles.

During the first 6 months of testing both the treated and untreated slabs showed evidence of spalling in the surface laitance layer. As the test continued the spalling increased to include the cement paste of the control slab, ultimately reaching a depth of 3/8 inch (9.5 mm). After the loss of the surface laitance the appearance of the top surface of the treated slab remained unchanged.

Figures 12 and 13 show the final appearance of the treated and control slabs, respectively. Figure 14 shows one of the vertical sides of the treated slab. The upper area which was protected by polymer was virtually unaffected while the lower untreated concrete experienced severe spalling 3/8 to 1/2 inch (9.5 to 12.7 mm) deep.

There was no evidence of delamination in either the control or treated test slabs.

4.3 Skid and Abrasion Resistance Test

The skid and abrasion resistance of surface impregnated bridge decks is of substantial interest to the highway industry. Numerous tests

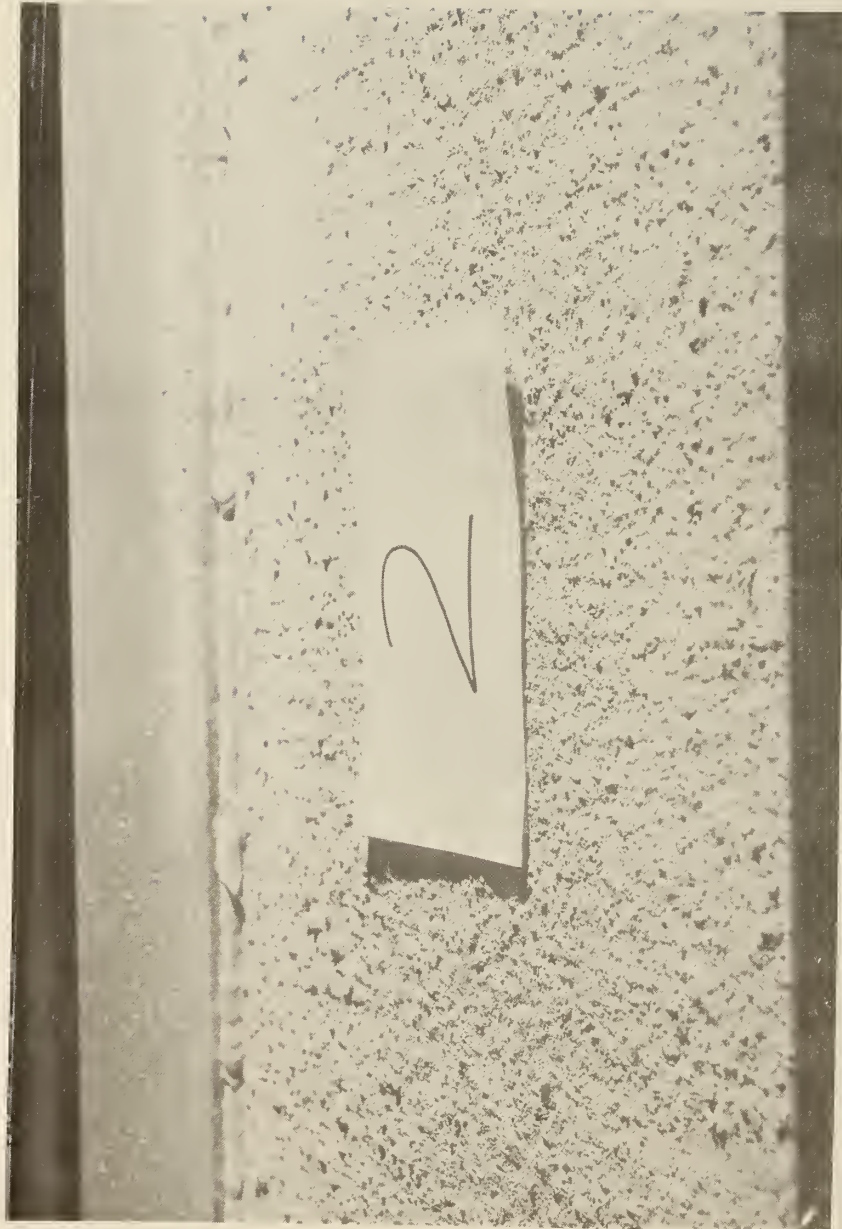


Figure 12. - Treated slab at completion of freeze-thaw test. Surface laitance spalled early in the test. Cement paste and aggregate were unaffected. Original broomed finish marks are visible.



Figure 13. - Control slab at completion of freeze-thaw test. Spalling extended 3/8 inch (9.5 mm) into the concrete. No evidence of the broomed finish remains.

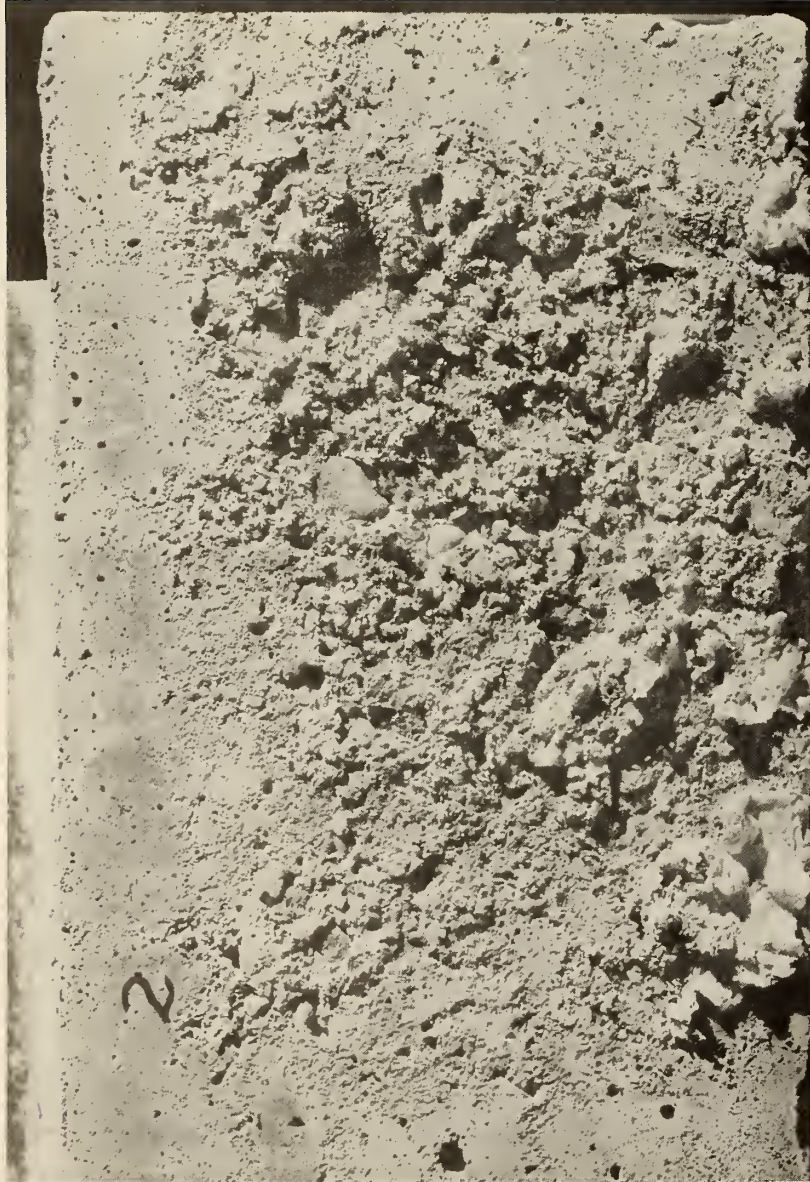


Figure 14. - Damage to the unimpregnated zone of the treated slab. This is a photograph of a vertical side of the treated slab. The damage began below the area protected by polymer. The impregnated upper zone of the concrete was undamaged.

have been performed on fully impregnated concrete to determine its resistance to abrasion. The results of these tests indicate that PIC displayed increased resistance to abrasion over that of conventional concrete. The surface impregnation process was, therefore, not expected to adversely affect the abrasion resistance of concrete. There was concern, however, that the inclusion of a hard, glassy polymer in the wearing surface of a concrete deck might result in lowered skid resistance as the surface became worn and polished by wheeled traffic.

A series of skid and abrasion resistance tests was performed for the Bureau by the Central Laboratory of the Maryland State Highway Administration using their circular test track facility. Two types of specimens were tested: untreated controls and surface impregnated test specimens. With each type there were three surface finish variations:

1. A rough broomed finish
2. A slightly polished finish prepared by grinding the broomed finish until the broom marks just disappeared
3. A fully polished surface, ground smooth prior to testing

The concrete used to cast these specimens contained 3/4-inch (19-mm) maximum size aggregate and type I cement. Mix data include:

Cement factor - 6.5 bags per cubic yard
Water-cement ratio - 0.46
Entrained air - 6 percent

The test concrete was cast, surface treated by the Bureau, and shipped to Maryland for testing. Polymer impregnation was accomplished using the saturated sand technique with the 95-5 MMA-TMPTMA monomer system catalyzed with 1 percent AMVN and polymerized in a forced draft oven at 158°F (70°C). Prior to impregnation the concrete was oven-dried at 250°F (121°C) for an overnight period. Polymer penetration was 1-3/4 inches (44 mm) deep.

Two series of track tests were required. In series A the performance of broomed finish and slightly polished impregnated specimens was compared with broomed finish and slightly polished control specimens. In series B the performance of broomed finish and fully polished impregnated specimens was compared with broomed finish and fully polished untreated controls. The specimens of test series A were exposed to 1,100,000 wheel passes. Those of series B were exposed to 1,400,000 passes.

During these tests four types of data were taken at periodic stops:

1. British Portable Tester, ASTM: E 303. - A measure of surface friction properties.

2. Strain. - A Maryland experimental process of determining surface friction properties based on the strain experienced by a counter rotating wheel driven over wet track test specimens.

3. Wire Shadow. - A Maryland experimental method of determining changes in surface texture based on measurement of shadow areas of sequential photographs of the test specimens.

4. Sand Patch. - A method of determining changes in surface texture based on the weight of sand required to fill the surface irregularities.

Based on these data it was concluded that the surface impregnation treatment had essentially no effect on skid resistance or on the rate of polishing of the treated concrete. The detailed report of these studies as prepared by the Maryland State Highway Administration is included in appendix A.

5. FIELD TESTS

The final objective of this research program was to surface impregnate a full-size bridge deck. However, in order to evaluate the treatment process and materials developed during the laboratory tests, two preliminary field trials were performed prior to the full-scale bridge treatment. The first of these trials involved the treatment of a 15-foot (4.6-m) by 20-foot (6.2-m) section of a 7-inch (178-mm) thick concrete slab on grade. In the second field trial, one 10-foot (3.0-m) wide lane of a 30-foot (9.1-m) long concrete bridge deck located on the Denver Federal Center was treated.

5.1 Concrete Slab on Grade

The concrete slab selected for this test was approximately 15 feet (4.6 m) wide by 30 feet (9.1 m) long with a nominal thickness of 7 inches (178 mm). This slab was nonreinforced and had been placed approximately 1 year prior to the treatment.

The basic procedure for the surface impregnation treatment involved drying with hot forced air, impregnation with the 95-5 MMA-TMPTMA monomer system using the saturated sand technique and hot forced-air polymerization.

To contain the heat for drying and polymerization, a 15-foot (4.6-m) by 30-foot (9.1-m) by 2-foot (610-mm) high portable enclosure was constructed over the concrete area to be treated. This enclosure consisted of an aluminum frame covered with 1/2-inch (13-mm) thick fiber building board insulation on its top and sides. The building board was covered with polyethylene film to provide weather protection.

Galvanized sheet metal ducts 12 inches (305 mm) square were located on the outside of the enclosure along each 30-foot (9.1-m) side. These ducts contained standard heat registers on 3-foot (914-mm) centers to deflect hot air into the enclosure. Hot air was provided by a 250,000-Btu (73.3-kW) gas-fired space heater attached to each duct. The hot air was exhausted from the enclosure at the bottom of the vertical end panels. These heaters had electrically powered blower fans. Temperature control within the enclosure was accomplished with a proportioning thermistor controller which cycled the heaters as needed. Thermocouples were attached to the concrete surface and used to monitor temperature throughout the treatment.

The planned treatment schedule was as follows:

1. Drying for 3 days (60 hours) at 225° to 250°F (107 to 121°C) surface temperature

2. 1-day cooling
3. Monomer application with overnight soak
4. Polymerization at 140° to 160°F (60° to 71°C) for 6 hours

The drying cycle was started on June 19, 1974. Immediately after start of the drying cycle one of the heaters malfunctioned. It was felt that the remaining heater would provide sufficient heat; however, the single heater did not have sufficient capacity and after 3 days the maximum concrete surface temperature was only 170°F (77°C). The height of the enclosure was reduced to 18 inches (457 mm) resulting in a surface temperature increase to 180°F (82°C). Next, the enclosure size was reduced to 15 feet (4.6 m) by 20 feet (60.9 m) and the desired concrete surface temperature was thus obtained. The concrete was dried for an additional 3 days at 225° to 250°F (107° to 121°C). The heat was then turned off and the concrete permitted to cool. Heat had been applied to the concrete for about 8 days with only several hours cooling between enclosure modifications. During this long heat application the ground beneath the slab had been heated and 3 days of cooling were required before the concrete surface temperature dropped below 90°F (32°C).

When this surface temperature was reached, the enclosure was removed and a 1/4- to 3/8-inch (6.4- to 9.5-mm) deep layer of dry sand was placed over one-half of the dried concrete surface. The remaining half of the area was not treated to serve as a control for future evaluation. A monomer system composed of 95-5 MMA-TMPTMA, catalyzed with 1/2 percent AMVN, was applied to the sand until full saturation was achieved. The monomer-saturated sand was then covered with polyethylene film and left to soak overnight. The following morning the sand appeared dry and additional monomer was applied to the sand until a damp but not saturated appearance was achieved. The total monomer application rate was 1.5 lb/ft² (7.33 kg/m²). The cover was replaced on the enclosure, and with both heaters operational, the concrete surface was quickly raised to 160°F (71°C) and maintained at that temperature for 6 hours to polymerize the monomer.

After polymerization, the enclosure was removed and two 2-inch (51-mm) diameter cores were taken from the concrete and split for inspection. Polymer penetration was 1/2 to 5/8 inch (12.7 to 15.9 mm) with no evidence of surface evaporation. Several small areas of the treated surface contained a lightly bonded composite of polymer and sand. The shallow polymer penetration in this test may have resulted from moisture migration back into the concrete during the long cooling cycle. The exact reason for such shallow penetration was not established.

5.2 Denver Federal Center Bridge

A small concrete bridge of unknown age and history located on Center Street on the Denver Federal Center was chosen for the second field trial. This bridge carries a heavy volume of traffic during a normal workday. Permission to treat the bridge was obtained provided one lane remained open to traffic. The west bound traffic lane was selected for surface impregnation and the east bound lane, serving as the control, remained open to traffic.

The enclosure used to heat the slab on grade was modified to cover a concrete surface area 12 feet (3.6 m) wide by 30 feet (9.1 m) long and installed on the Center Street bridge. Electrical power was not available at the bridge, so a portable generator was put into service to provide power for the heaters, the temperature recorder, and the temperature control system. A 500-gallon (1,892-ℓ) propane gas tank was located at the bridge to supply gas for the heaters. Figure 15 shows the enclosure and control equipment in operation on the Center Street bridge.

As in the previous test with this equipment the heat transfer ducts were located on the exterior of the enclosure. Surface temperature was monitored with six thermocouples located on the concrete surface. Two additional thermocouples located 1 and 2 inches (25 and 51 mm) deep into the concrete at the approximate center of the heated area provided data on the temperature conditions within the concrete deck. A thermistor temperature controller was used to cycle the heaters as needed to reach and maintain the selected concrete temperatures.

The drying cycle was begun on July 15, 1974, but afternoon rain showers repeatedly interrupted the process until July 24. During these showers runoff from the street above the bridge seeped under the enclosure. Simple diversion dams would have eliminated this problem but would have required closing the bridge to all traffic.

Beginning on July 24, the weather cleared sufficiently to obtain a 3-day drying cycle at 225° to 250°F (107° to 121°C). The drying cycle was completed at about 6 p.m. the third day and the heat was turned off to allow the deck to cool. Twenty-four hours later the concrete had cooled sufficiently to permit monomer application but because of threatening weather the impregnation cycle was postponed until the afternoon of the following day.

Late in the afternoon of July 29, a layer of dry sand about 3/8 inch (9.5 mm) thick was spread over the dried concrete and saturated with a monomer system composed of 95-5 MMA-TMPTMA catalyzed with 1/2 weight percent AMVN. The monomer was left to soak overnight. The

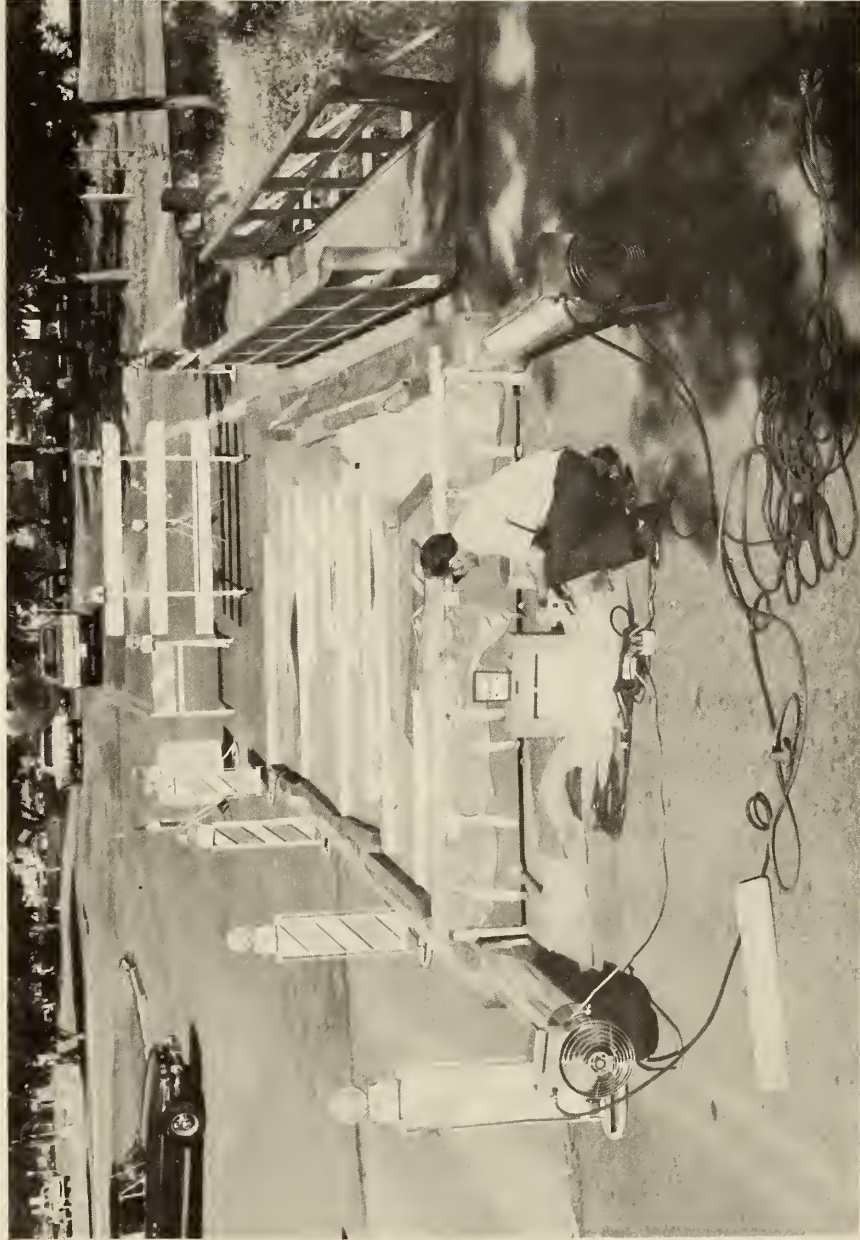


Figure 15. - Drying enclosure on Center Street bridge. Heaters connected to external sheet metal ducts. Temperature recorder and thermistor controller located between space heaters.

following morning the sand was resaturated with monomer and allowed to soak for 2 additional hours while the heating enclosure was reinstalled over the bridge. The total monomer application rate was 1.7 to 1.8 lb/ft² (8.3 to 8.8 kg/m²).

The heat ducts and heaters were located in their proper position and the heater fans turned on to ventilate monomer vapors as the enclosure cover was being installed. This eliminated the possibility of an explosive vapor-air mixture within the enclosure at the moment the heat was turned on.

After the enclosure was installed and ventilated, the heater burners were turned on and the concrete surface temperature increased to 120°F (49°C) over a 5-hour period. This relatively slow heat-up rate was used so that excess monomer would evaporate from the sand prior to polymerization and prevent the problem of polymer bonded sand which was experienced with the slab on grade. The concrete surface temperature was then increased and maintained between 130° and 160°F (55° and 71°C) over a 3-hour period to complete polymerization.

After polymerization the heating enclosure and equipment were removed from the bridge. No sand was bonded to the concrete. Two 2-inch (51-mm) diameter cores were taken to determine polymer penetration. When these cores were split for inspection, a polymer penetration of 2 inches (51 mm) was found in each specimen. One core, however, showed evaporative monomer loss from the surface of the concrete. This apparently polymer-free zone extended approximately 1/2 inch (13 mm) below the riding surface of the deck. A polymer impregnated zone of concrete extended approximately 1-1/2 inches (38 mm) below this boundary. By comparing the location of this core with the surface temperature records it was found that this core was from an area of relatively high surface temperature, in excess of 170°F (77°C), during the polymerization cycle.

Two additional cores were taken from treated areas that contained vertical cracks prior to treatment. In one core, polymer bonded the cracked concrete together while in the other it appeared to have no significant effect.

In order to determine the properties of the concrete prior to treatment, test cores were taken from several areas in the untreated traffic lane. Tests on these cores yielded the following data:

Compressive strength	2,200 lb/in ² (15.2 x 10 ⁶ N/m ²)
Specific gravity	2.34
Unit weight	145.8 lb/ft ³ (2.335 kg/m ³)
Percent air voids	6.31 based on dry weight

From the results obtained in the two field trials it appeared feasible to proceed with the selection of a bridge for the full-scale field demonstration test.

5.3 Quincy Avenue Bridge (near Cherry Hills Village, Colorado)

Four basic requirements were used to select the bridge for the field demonstration:

1. The bridge must not have been opened to traffic or previously treated with deicing salts, linseed oil, or other chemicals.
2. The bridge deck must be constructed of conventional portland cement concrete and water cured (no curing compounds).
3. In order to complete the surface impregnation treatment prior to the beginning of winter weather the bridge deck should have a construction completion date not later than the first of September.
4. Although not a rigid requirement it was desirable, for convenience, that the bridge be located within a 60-mile (96.6-km) radius of Denver.

Several bridges were investigated and rejected before a structure located in Cherry Hills Village near Denver was selected. This bridge, referred to as the Denver bridge, had a riding surface 28 feet (8.5 m) wide by 60 feet (18.3 m) long and was constructed of prestressed concrete box girders and a nominal 7-inch (177-mm) thick cast-in-place deck. The concrete for the deck was placed on September 17, 1974, and moist cured until October 1, 1974, when the drying cycle was begun. These times were somewhat later than desired.

A larger heating enclosure was designed and fabricated and four 500,000-Btu (146-kW) space heaters were obtained. Two heat ducts were used as before but were located within the enclosure to reduce heat loss.

The temperature monitoring and control system was similar to that used in the field trials but the electric power requirements of the four larger heaters exceeded the capacity of the controller and a relay system was thus used for on-off cycling.

A temperature recorder and eight thermocouples were used to monitor concrete temperatures at six surface locations and at 1- and

2-inch (25- and 51-mm) depths in the concrete. Propane gas was provided by two 500-gallon (1,892-ℓ) tanks, one located at each end of the bridge.

Figure 16 shows the general layout of the electrical and gas systems that were used on the bridge.

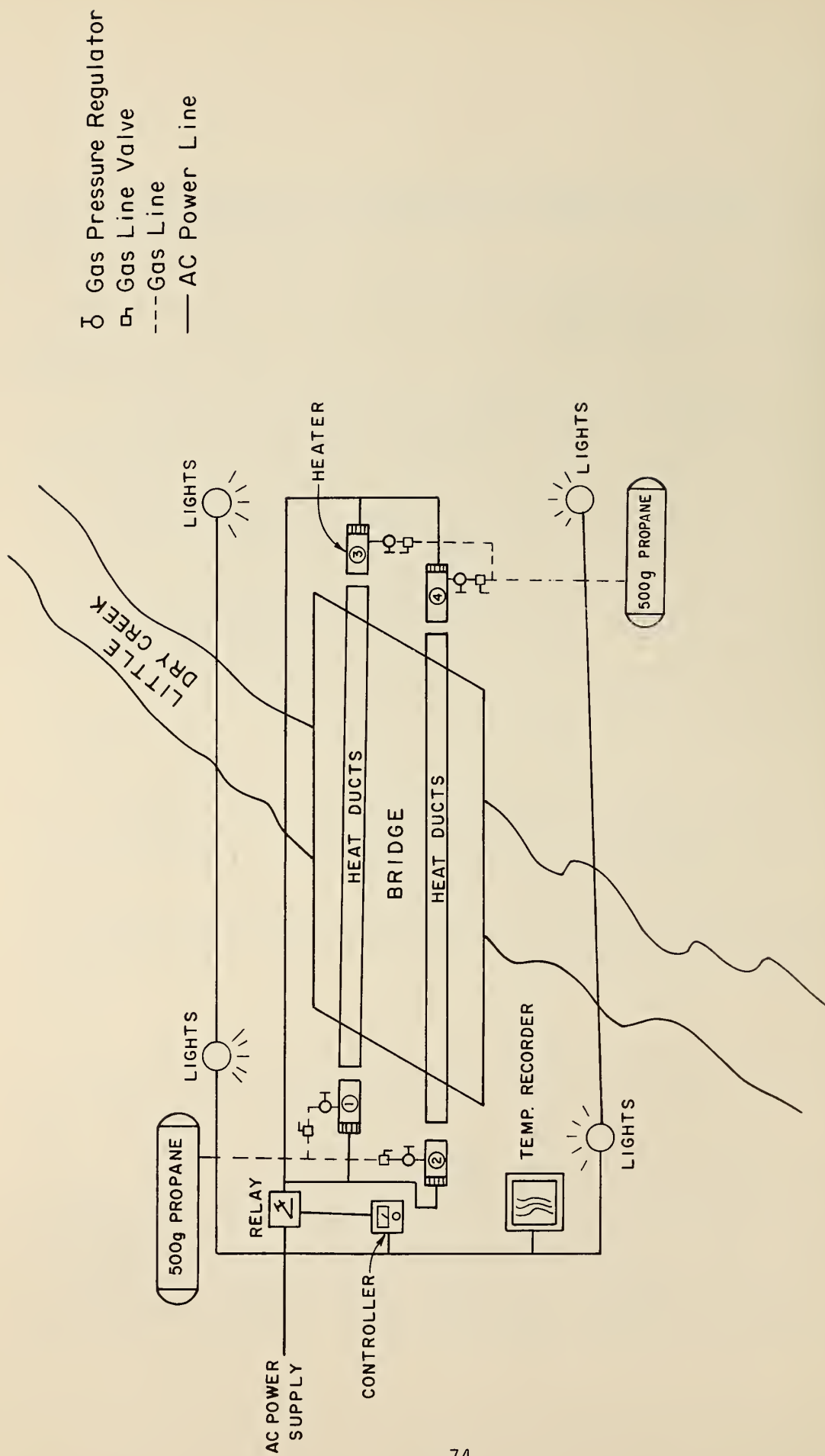
On October 1, 1974, the 30-foot (9.1-m) wide by 61-foot (18.6-m) long by 2-foot (610-mm) high heating enclosure with its various heating and temperature control equipment was installed on the bridge deck. Figure 17 shows the bridge at the time the sheet metal heating ducts were being installed. After the ducts were located, a prefabricated aluminum enclosure frame was constructed. This frame was built from 2-inch (51-mm) square tubing and was designed so that the horizontal members were connected to the vertical legs by slip joints. Figure 18 shows one section of the frame in place on the deck. The enclosure was divided into six such sections to permit rapid removal and reinstallation during the treatment process. Figure 19 shows the completed frame with heat ducts and heaters in place. The registers located in the heat ducts were adjustable in the vertical direction to permit even distribution of the hot air.

After the frame was in place the enclosure was covered with 1/2-inch (13-mm) thick fiber board insulation, figure 20, which was also used to form the vertical sides. The enclosure was then covered with a lightweight tarpaulin and polyethylene sheeting for weatherproofing as shown in figure 21.

Figure 22 shows the thermocouple temperature recorder and the proportioning controller that was used to maintain the selected drying and polymerization temperatures.

The complete enclosure in operation is shown in figure 23.

The drying heatup cycle was begun at 5 p.m. October 1 and continued until the concrete surface temperature reached 225° to 250°F (107° to 121°C) at about 5 a.m. on October 2. During this period it was necessary to adjust and vary the gas pressure to the individual heaters to maintain temperature uniformity over the concrete surface. A problem was encountered due to the cooler nighttime temperatures which fell below freezing several times during the process. These low temperatures in combination with the rapid rate of propane withdrawal from the tanks produced very low tank temperatures and a corresponding loss of propane pressure during the period when it was most needed. Figure 24 shows the ice that formed on one of the propane tanks during the first night.



GENERAL LAYOUT OF QUINCEY AVENUE BRIDGE EQUIPMENT
FIGURE 16



Figure 17. - Locating heat ducts on bridge deck. Bridge has a walkway on the far side and a bridle path on the near side.



Figure 18. - Section of enclosure frame on bridge deck.



Figure 19. - Completed enclosure frame with heaters and heat ducts on deck.

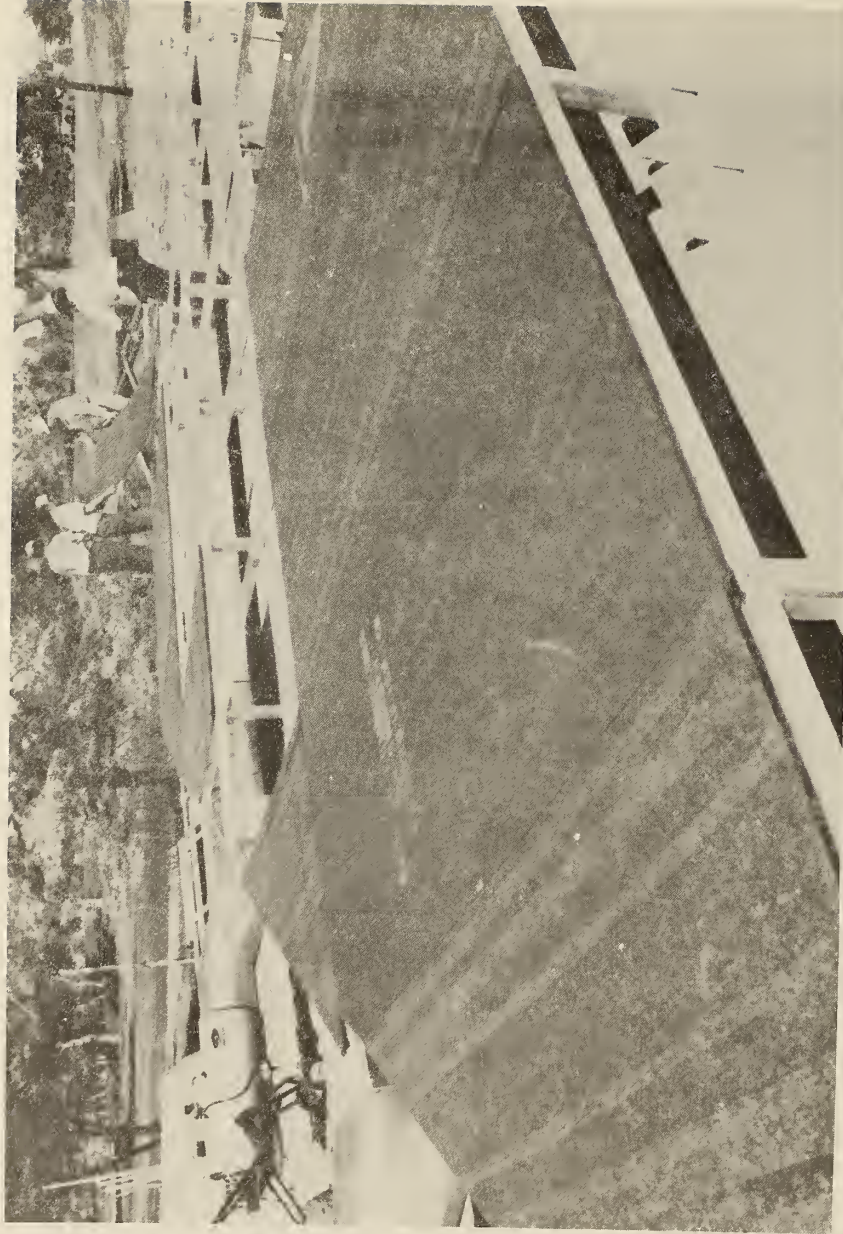


Figure 20. - Fiber board cover being placed on enclosure frame.



Figure 21. - Weatherproofed enclosure. Lightweight tarpaulin and polyethylene plastic have been placed over fiber board cover.



Figure 22. - Temperature recorder and controller. Temperatures from eight locations were monitored with thermocouple recorder. Small thermistor controller sitting on recorder provided data on air temperature within the enclosure and activated a relay system to control heater cycles.



Figure 23. - Drying enclosure in operation. Hot air from the heaters was exhausted from adjustable vents at the bottom of the end panels. Variation of the vents provided a measure of temperature control.



Figure 24. - Propane tank after first night of drying. Cooling due to rapid gas withdrawal and low nighttime temperatures caused the tank to ice and resulted in lower gas supply pressures.

Several remedial steps were taken to improve this situation including reducing the hot air exhaust area at the vertical ends of the enclosure and reducing the airflow through the heaters. It was determined that there was no safe method of heating the propane tanks with the equipment on hand. These efforts improved the rate of heatup but were only partially successful. The heatup cycle required about 12 instead of the expected 8 hours.

After the desired surface temperature was obtained, heater gas pressures were reduced and adjusted to balance the system and provide continuous operation of the heaters. During the drying cycle it is estimated that the heaters were operated at about one-half of the rated capacity.

The 225° to 250°F (107° to 121°C) surface temperature was maintained for about 37 hours until 6 p.m. on October 3. At that time, several expansion cracks were noticed in the unheated sidewalk concrete adjacent to the riding surface of the bridge deck. Although it had been anticipated that these harmless cracks would form in this area, a conservative approach was taken and the concrete surface temperature was reduced to 175° to 212°F (79° to 100°C). This temperature range was then maintained for 23 hours until the drying heat was turned off at 5 p.m. on October 4. The bridge was then allowed to cool overnight with the enclosure in place. During this cooling cycle the nighttime low temperature was 30°F (-1°C).

The following morning, October 5, the enclosure and the various heating and temperature control equipment, with the exception of the temperature recorder which was used to monitor concrete temperature, were removed from the bridge deck. Figure 25 shows several of the 20-foot (6.1-m) by 16-foot (4.9-m) frame sections after enclosure removal. As can be seen in this figure, the loosely fitting joints allowed the frame to conform to surface irregularities far in excess of those acceptable for bridge deck riding surfaces.

A layer of dry sand, 1/4- to 3/8-inch (6.4- to 9.5-mm) thick, was spread uniformly over the dried concrete surface, figure 26. The sand was then covered with a full-size polyethylene sheet to protect the dried concrete while the equipment and materials were prepared for monomer impregnation, figure 27.

The monomer system used to impregnate the bridge was composed of 95-5 MMA-TMPTMA with 1/2 percent AMVN catalyst. Seven 55-gallon (208-ℓ) drums of this monomer system were mixed the previous day using equipment at the laboratory impregnation facility. After the monomer was mixed it was stored overnight in a refrigerated storage area at -20°F (-29°C) and transported to the bridge site

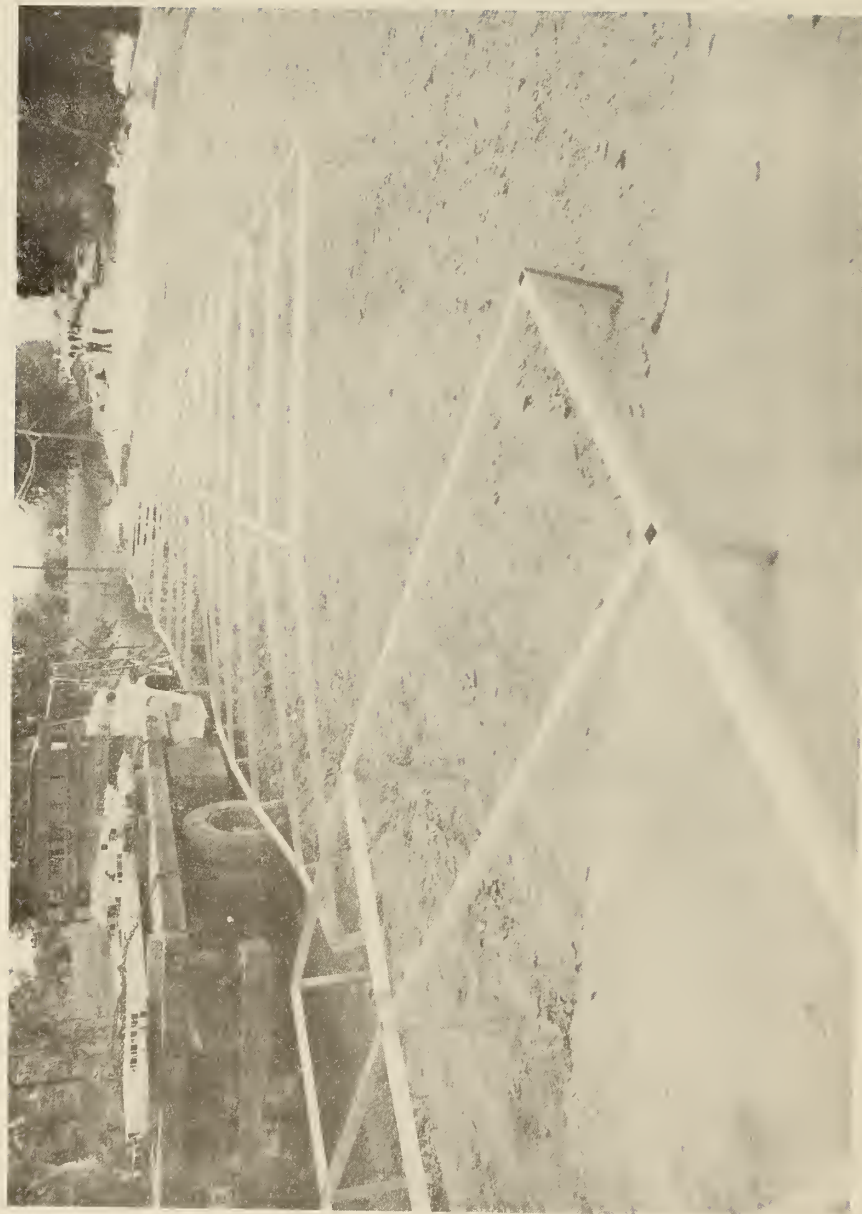


Figure 25. - Freestanding enclosure frame sections. These sections eliminated the need to completely dismantle the frame for removal and reinstallation.



Figure 26. - Spreading sand on the deck. Spreader had hardened steel pins on the bottom to maintain proper sand depth.

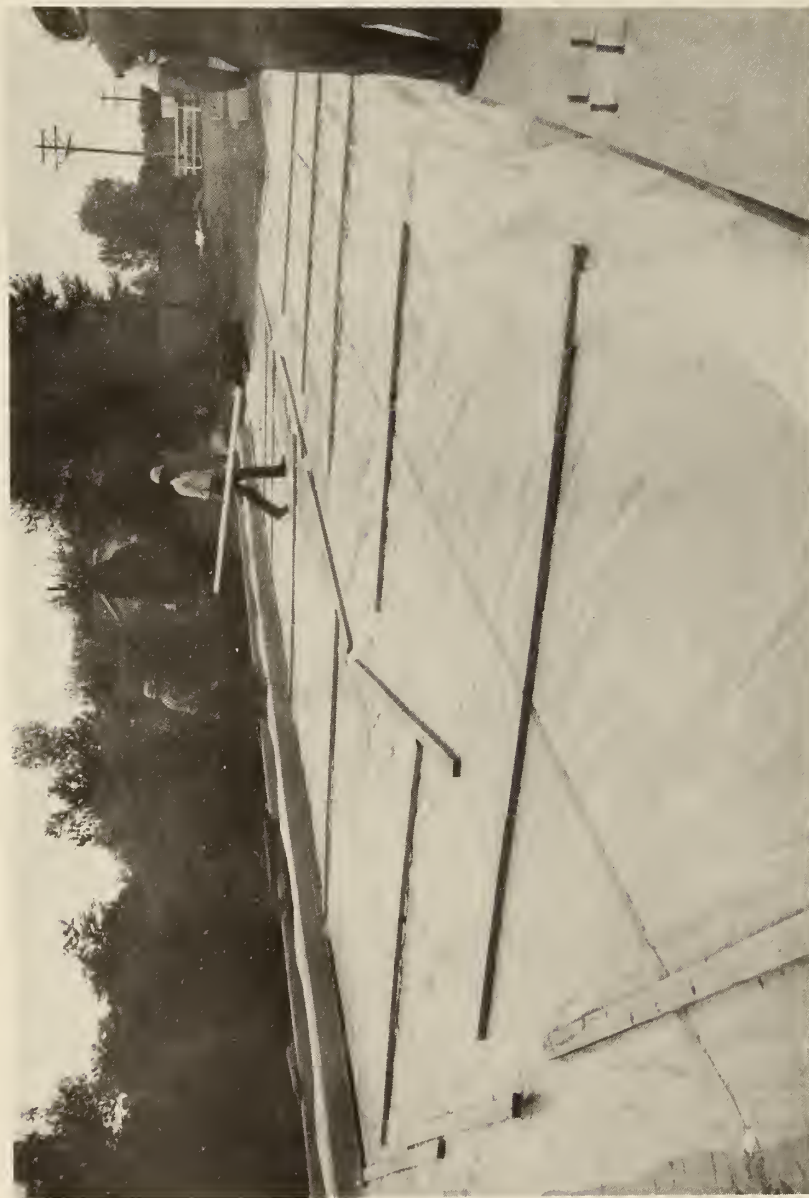


Figure 27. - Polyethylene sheet on deck. This plastic cover was placed after the sand was spread to provide weather protection and in preparation for monomer application.

early in the afternoon of October 5. Note: (Under normal conditions catalyst addition would have been made just prior to use on the bridge deck. It was felt, however, that the catalyzed monomer could be safely stored at the very low temperature of -20°F (-29°C) for an overnight period and catalyst mixing was performed at the laboratory facility for convenience.) Monomer temperature at the time of catalyst addition on October 4 was approximately 55°F (13°C). During transportation to the bridge site and while at the bridge site prior to use, the monomer drums were covered with white tarpaulins to reduce the effects of solar heating. A safety check of the bridge site was made to remove sources of ignition that could create a fire hazard. Warning signs were posted and "No Smoking" rules were enforced. Chemical fire extinguishers were placed at the corners of the bridge.

Monomer was applied to the deck using a small rubber-wheeled cart containing a monomer drum and a 14-foot (4.3-m) wide spray bar. Monomer drums, which weighed about 500 pounds (227 kg), were loaded onto the cart with a simple tripod hoist, figure 28.

The first application of monomer was begun just prior to sunset to eliminate the possibility of solar radiation causing premature polymerization of the monomer-saturated sand. The polyethylene plastic sheet covering on the sand was folded away from one lane of travel on the bridge and the uncovered area from the centerline to the curb was saturated with monomer. This was accomplished with a two-way pass of the monomer cart, figure 29. The polyethylene was replaced, figure 30, to prevent evaporative monomer loss and reduce fire and odor problems. Monomer then was applied to the remaining lane of traffic. Figure 31 shows the appearance of the second lane of traffic just at the completion of monomer application. The polyethylene was replaced over this area and the monomer was allowed to soak into the concrete for about 4 hours.

During this first application 210 gallons (795 ℓ) of monomer were applied at a rate of approximately 1.1 gal/yd^2 ($5.0 \text{ } \ell/\text{m}^2$). On a weight basis the application rate was very nearly 1 lb/ft^2 (4.9 kg/m^2).

After the 4-hour soak period a second monomer application was made. Approximately 110 gallons (416 ℓ) or 0.5 lb/ft^2 (2.4 kg/m^2) of monomer were required to saturate the sand during this application. This monomer was allowed to soak into the concrete overnight. The following morning, October 6, the sand appeared slightly dry for polymerization and a third monomer application, 55 gallons (208 ℓ) was made. A total of 375 gallons (1,419 ℓ) of monomer was applied to the concrete at a rate of 1.7 lb/ft^2 (8.5 kg/m^2). This rate later prove to be slightly excessive.



Figure 28. - Tripod-hoist used to load monomer cart. Full monomer drum weighed approximately 500 pounds (227 kg).



Figure 29. - Applying monomer. Gravity-fed spray bar applied monomer to one lane of travel. Cart in this photograph is making second application pass over the sand. Polyethylene has been folded back to expose area to be treated.



Figure 30. - Polyethylene replaced to reduce monomer evaporation.



Figure 31. - Monomer saturated sand on deck. This is the desired appearance of the fully saturated sand. Photograph was taken just at the completion of monomer application to the second lane of travel.

The enclosure frame, heat ducts, heaters, and the temperature monitoring and control equipment were then relocated over the treated, polyethylene-covered sand and concrete. The heater fans were turned on to ventilate monomer vapors while the fiber board cover and sides were placed.

Heat application for polymerization started early in the afternoon of October 6 and the concrete surface temperature was slowly increased to 140°F (60°C) through a 3-1/2-hour period. This temperature was maintained for about 2-1/2 hours and the heaters were then turned up to increase the surface temperature to 180°F (82°C). This higher temperature was maintained for 12 hours to complete the polymerization cycle and insure that no unpolymerized monomer was left in the concrete.

The heat was turned off and the enclosure removed from the deck early in the morning of October 7.

Inspection of the treated surface after removal of the loose sand indicated that approximately 5 to 7 percent of the roadway surface was covered with sand that was bonded to the concrete by polymer. Figure 32 shows an area affected by this material. The bonded sand did not create structural or riding quality problems but gave an unsightly appearance. For this reason it was subsequently removed with equipment similar to that used to groove airport runway surfaces. Figure 33 shows the completed bridge after it had been opened to traffic.

Eleven 2-inch (51-mm) diameter cores were drilled from the treated concrete deck. Seven of these were broken apart in tensile splitting to provide a freshly broken surface for inspection. Polymer penetrations of 1 to 1-1/2 inches (25 to 38 mm) deep were evident in these cores with no evidence of surface evaporation loss. Figure 34 shows the dark polymer penetration that was common to these cores. The cracks in the photograph are the result of the tensile splitting technique of breaking the cores.

Two of the remaining cores were sawed into 2-inch (51-mm) diameter by 1-inch (25-mm) high disks and tested to determine their water absorption properties. These data are listed in table 14. From the test results it can be seen that the water absorption in the treated zone is roughly one-third that of the lower untreated concrete. It should be noted, however, the water absorption properties of this concrete are rather high. Normally our untreated laboratory concrete runs about 6.5 percent by dry weight and 1 to 3 percent after polymer impregnation using the surface impregnation techniques.



Figure 32. - Sand bonded to deck. This material was subsequently removed by grinding.



Figure 33. - Completed bridge after treatment, November 1974.

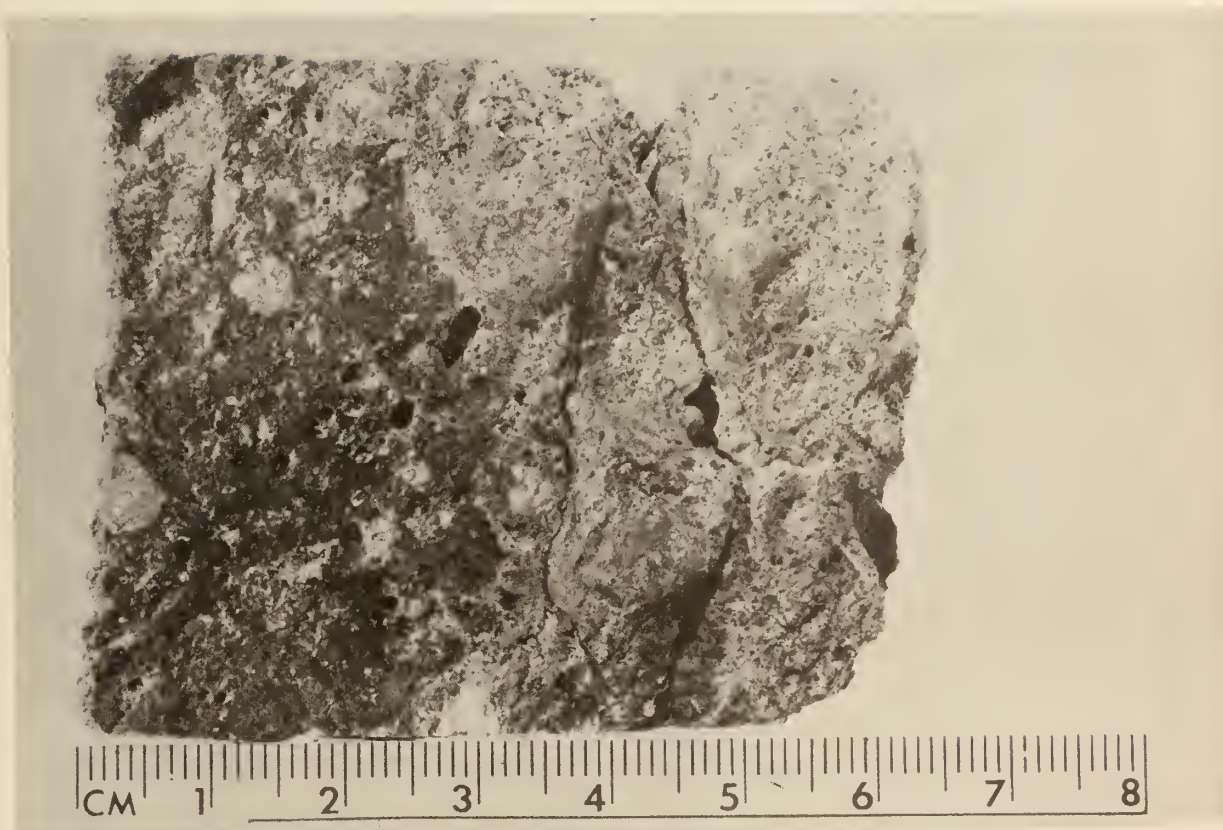


Figure 34. - Polymer penetration seen in core sample. The polymer appears as a dark zone at the top of the core. Cracks in the bottom of the core were created by the tensile splitting technique used to break the core.

Table 14

WATER ABSORPTION TEST OF QUINCY AVENUE CORES
(ASTM No. C 497 - 5-hour Boiling Water)

Core No.	Depth below surface (inches)	Water absorption (percent of dry weight)	Unit weight (lb/ft ³)
1	0-1 (visible polymer)	3.1	141.0
1	1-2	7.5	139.7
1	2-3	9.2	140.3
1	Bottom 5-6	8.9	138.9
2	0-1 (visible polymer)	2.8	141.2
2	1-2	8.9	139.2
2	2-3	9.8	139.2
2	Bottom 5-6	9.8	138.1

During an inspection trip to the bridge several weeks after the surface impregnation treatment it was observed that the treated deck surface was covered with roughly circular hairline pattern cracks. These cracks were quite obvious because the deck was drying from a recent rain shower and the water wet cracks appeared darker than the concrete, figure 35. The cracks were not noticed prior to bridge treatment nor were they noticed immediately after treatment.

Additional cores that included these cracks were taken from the deck and subjected to various microscopic examinations and petrographic tests. The cracks were found to extend approximately 2-1/2 to 3 inches (64 to 76 mm) into the concrete. Polymer was present at the fracture plane but the cracks were not completely sealed with polymer. The concrete was sound and there was no evidence of any type of adverse chemical reaction. These tests ruled out a number of potential causes of the cracking such as sulfate reaction or adverse cement-aggregate reaction, but did not identify the actual cause. It was suspected, however, that the early concrete age (14 days at the time of drying) was a contributing factor. The concrete of the two previous field trials was inspected. One small area of similar cracking was found on the Center Street Bridge in the treated lane. Another area was found on the untreated lane - far removed from the effects of drying heat, impregnation, or polymerization. The slab treated in the first field trial contained pattern cracking in the concrete at one corner adjacent to an expansion joint.

Several treated laboratory specimens, including three 2-foot (610-mm) by 6-foot (1.8-m) by 7-inch (180-mm) thick reinforced slabs, were inspected. None were cracked. In an attempt to create the cracks in the laboratory a 14-day-old 4-foot (1.2-m) square concrete slab was taken from the fog curing room and placed in a preheated 400°F (204°C) drying oven for 18 hours. Inspection of the surface indicated that no cracking had occurred. From the above studies, it was concluded that the cause of cracking of the bridge deck concrete or the time at which the cracking occurred could not be determined. Additional information would have to come from observation of future bridge treatments.

5.4 Additional Field Treatments

During the summer of 1975 a cooperative agreement between the Bureau and the Federal Highway Administration's Office of Development, Implementation Division, was formalized. Included in the objectives of this agreement was the provision that the Bureau provide technical assistance to FHWA during the surface impregnation treatment of several concrete bridge decks. A complete discussion of the implementation activities will be included

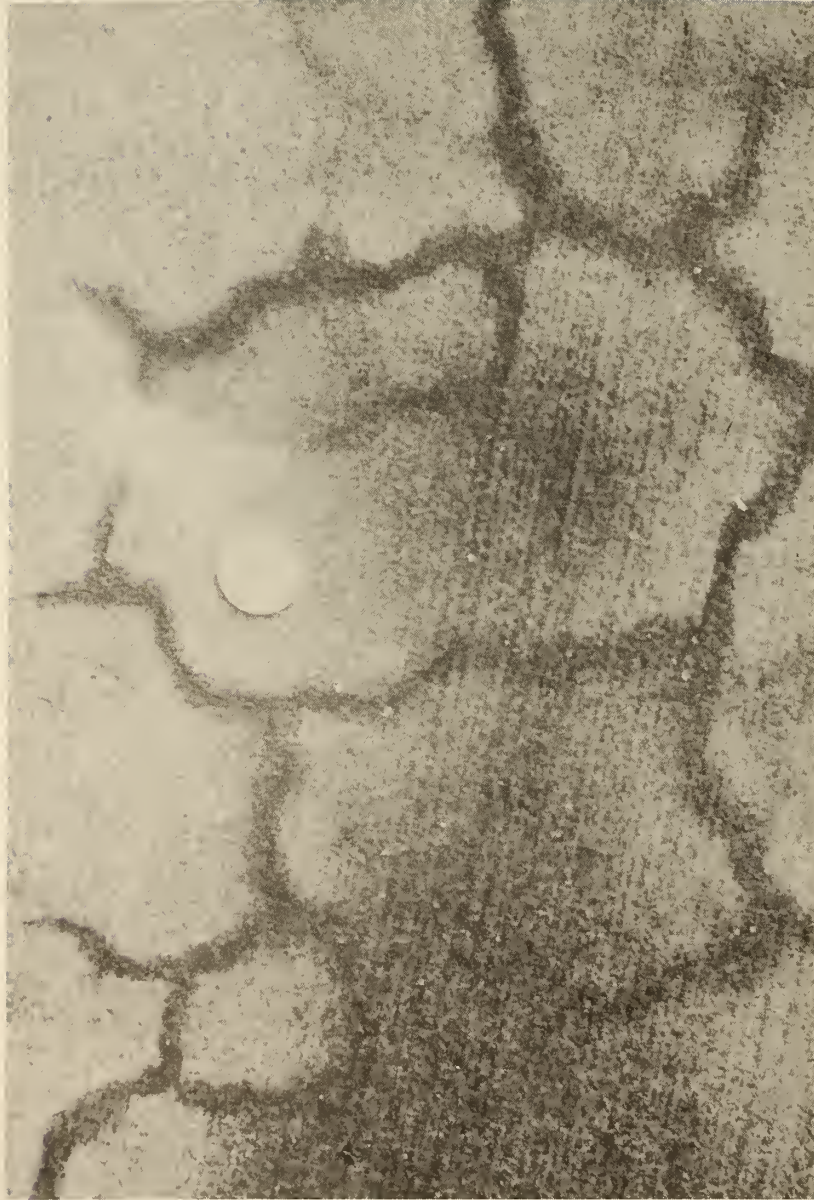


Figure 35. - Water wet cracks in surface of treated bridge deck.

in a later publication. However, because of the close relationship between the research efforts and the implementation effort, a brief discussion of the first two field evaluations in the implementation program is included in this report.

5.4.1 Idaho Bridge Treatment

The second full-size bridge to be treated with the surface impregnation process was located in East Hope, Idaho, on Lake Pend Oreille. This bridge was 38 feet (11.6 m) wide by 50 feet (15.2 m) long with a cast-in-place deck a nominal 7 inches (178 mm) thick. The riding surface of the bridge had 5 percent superelevation. The surface impregnation treatment was performed by the bridge construction contractor under a contract with FHWA and with technical assistance provided by the Bureau.

The treatment process was basically similar to that used on the Quincy Avenue Bridge. There were, however, several important differences:

The deck concrete was water cured for 28 days and allowed to air dry for about 2 weeks prior to beginning the drying process. Inspection of the concrete indicated that it was free of cracks prior to treatment.

The drying and polymerization heating enclosure was constructed of freestanding vertical supports covered with 4-foot (1.2-m) by 12-foot (3.6-m) fiberglass panels obtained from a rental company. Glass fiber insulation and polyethylene sheeting covered the enclosure. Figure 36 shows the bridge just prior to completion of the enclosure. The cover panels are stacked on the left parapet wall of the bridge ready for placement.

Three heating ducts with six 350,000-Btu (102-kW) heaters were used to provide and distribute heat over the deck.

The monomer system was composed of 95-5 MMA-TMPTMA with 1 percent AMVN catalyst by weight. Two monomer applications were made 4 hours apart. The total monomer application rate was 1.5 lb/ft² (7.3 kg/m²).

The drying heatup cycle was begun on July 23, 1975. Due to various mechanical and design problems with the heating system, heating continued for 26 hours until the surface of the deck reached a temperature of 250°F (121°C). The drying cycle was continued at this temperature for an additional 60 hours. Total heat application time for drying was 86 hours. A close inspection of the deck surface at the completion of the drying cycle and before the concrete had cooled



Figure 36. - Enclosure used on Idaho Bridge. Simple support system and rental panels made enclosure installation and removal very convenient. Three heat ducts and six heaters were used with this enclosure.

revealed the presence of hairline pattern cracking similar to that on the Quincy Avenue Bridge.

After cooling, monomer was applied using a cart as shown in figure 37. This cart was fitted with adjustable spray bar supports as shown in figure 38 to permit the spray bar to be leveled horizontally on the superelevated deck.

Impregnation and polymerization cycles were performed similar to the treatment used on the Quincy Avenue Bridge and resulting polymer penetrations were 1-1/4 to 1-3/4 inches (32 to 44 mm). Approximately 5 percent of the deck was covered by bonded sand which was removed by chipping and grinding. The cracks that formed during the drying cycle had not opened during the impregnation and polymerization cycles and, as before, polymer was found at the crack fracture plane but the cracks were not sealed by the polymer. Figure 39 shows the bridge deck prior to removal of the bonded sand.

In reviewing the results of the two bridge treatments it was felt that the conservative 250°F (121°C) 60-hour drying cycle was excessive. All of the polymer penetrations exceeded the desired 1-inch (25-mm) depth. The long drying cycle may have contributed to the pattern cracking problem. A series of four 2-foot (610-mm) square concrete slabs were surface impregnated in the laboratory with 24-hour-long, 250°F (121°C), drying cycles. Polymer penetrations of 1 to 1-1/4 inches (25 to 32 mm) were achieved.

It also appeared that the 1.5 lb/ft² (7.3 kg/m²) monomer application rate was slightly excessive. A reduced rate would be used on the next field project.

5.4.2 California Bridge Treatment

The third full-scale bridge treatment was performed in October of 1975 on a bridge over Rattlesnake Creek on State Route 36 near Forest Glen, California. This bridge is 211 feet (64.3 m) long with a deck width of 33 feet 8 inches (10.3 m). The cast-in-place concrete deck is 8 inches (203 mm) thick with superelevation varying from 0 to 6 percent. Surface impregnation was performed by the bridge construction contractor under contract with FHWA and with technical assistance from the Bureau. Because of the length of this bridge it was treated in separate sections, each approximately 70 to 75 feet (21 to 23 m) long.

The drying and polymerization enclosure was identical to that used on the Idaho Bridge although it was only 22 inches (559 mm) high. This height reduction permitted more efficient heat transfer to the concrete deck.



Figure 37. - Monomer application cart and spray bar.



Figure 38. - Leveling supports on monomer cart. This system allowed the spray bar to be rotated to a horizontal position on the superelevated bridge deck.



Figure 39. - Sand bonded to Idaho Bridge. This material was removed by chipping.

Two heat distribution ducts were used with the four 500,000-Btu (146-kW) heaters from the Quincy Avenue Bridge.

The drying cycle was begun on October 14, 1975. A drying heatup period of 12 hours was required to raise the deck surface temperature to 250°F (121°C). This temperature was maintained for 24 hours before the heat was turned off. Inspection of the deck at the completion of drying (36 hours total heat application) revealed the presence of the hairline pattern cracks as seen on the two previous bridges.

Monomer was applied in two applications using the same monomer system and equipment as in Idaho. The two applications were made 6 hours apart at a total application rate of 1.4 lb/ft² (6.8 kg/m²). After the monomer had soaked overnight, the sand appeared to contain a slight excess of monomer. The enclosure was installed and the heat turned on to maintain a concrete surface temperature of 100° to 130°F (38° to 55°C) for about 3 hours to permit the excess monomer to evaporate. The surface temperature was then increased to 160° to 180°F (71° to 82°C) for about 12 hours to complete polymerization.

During cleanup operations, the impregnation sand was easily swept from the deck with only one very small area lightly bonded to the concrete. Polymer penetrations in nine test cores averaged 1-1/4 inches (32 mm) and the smell of water wet concrete was evident below the impregnated zone of the freshly broken test cores.

From these results it was determined that the drying cycle and monomer application rate could be further reduced for the subsequent treatment.

The next section of the bridge was treated similarly to the first but with the drying cycle reduced to 12-hour heatup followed by 12 hours at 250°F (121°C). The total monomer application rate was reduced to 1.3 lb/ft² (6.4 kg/m²).

The surface pattern cracking seen on the first bridge section was again apparent at the completion of the 12-hour drying cycle. Polymer penetrations varied from 3/4 to 1-7/8 inches (19 to 48 mm) with the 3/4-inch (19-mm) penetration occurring along the center of the bridge where drying temperatures were approximately 20°F (11°C) lower than the specified 250°F (121°C).

From these results it was concluded that a drying cycle of 12 hours at 250°F (121°C) - 24 hours total heat application - was still somewhat longer than necessary to achieve 1-inch (25-mm) polymer penetrations. It was also apparent that as the cycle time is

reduced, surface temperature uniformity becomes increasingly significant.

A more detailed discussion of the treatment process parameters and of investigations of the cracking phenomena are included in Section 6 - Recommended Treatment Process and Section 7 - Crack Formation and Treatment.

6. RECOMMENDED TREATMENT PROCESS

The surface impregnation process is undergoing continual development as the various laboratory and field tests are performed. The process as described should thus be considered as a basic guide to prospective users and subject to changes and refinements as experience in the field dictates.

The process was developed for and tested on new concrete. Its use on older concrete bridge decks exposed to extensive traffic, treated by other deck sealing methods, or contaminated with chloride has not been proven as this was not an objective of the program.

The durability tests reported herein were performed on specimens impregnated to a depth of at least 1 inch (25 mm), which the recommended surface impregnation process is designed to provide. Lesser penetrations would be achieved with shorter process cycles, since the drying and impregnation depths have been found to be functions of the square root of time. However, the durability effectiveness of penetrations less than 1 inch (25 mm) was not evaluated in this study.

6.1 Treatment Process

Within these limitations the recommended bridge deck surface impregnation process is as follows:

1. The concrete to be impregnated should be water cured and should have obtained a minimum compressive strength of 3,000 psi (21 MPa) prior to treatment. Generally, the use of curing compounds on concrete to be surface impregnated is not recommended unless tests are performed to prove that the curing compounds do not hinder the rate or depth of monomer penetration or contaminate the monomer and alter its polymerization characteristics.
2. The concrete to be impregnated should be inspected and cleaned, if necessary, to remove surface contaminants such as oils, tar, mud, construction debris, dirt, and trash. Contaminants such as oil, tar, or curing compounds may require sandblasting or steam cleaning. Normally, however, sufficient cleaning can be accomplished by sweeping or by blowing the deck clean with compressed air. Appropriate personnel safety precautions must be taken.
3. The concrete must be dried prior to impregnation. If drying is accomplished with hot forced air as described in this report the drying cycle should be considered as having

two parts. The heatup cycle should require 8 to 12 hours during which time the concrete surface temperature is uniformly increased to 250°F (121°C). This temperature should then be maintained for an additional 8 to 12 hours to complete the drying cycle. Thus, total heat application time for drying would be 16 to 24 hours.

Concrete surface temperatures should be monitored at several locations on the deck during drying to assure that uniform temperatures are being achieved. For this purpose, temperature variations over the deck surface greater than ±20°F (±11°C) is considered excessive.

If heating enclosures similar to those used on the Denver, Idaho, and California bridges are used for drying, their height above the concrete deck should be 24 inches (610 mm) or less to reduce temperature stratification above the concrete. With enclosures of this type it was found that heating at the rate of approximately 1,000 $\frac{\text{Btu}}{\text{hr}}$ per ft² of deck surface (3.2 kW/m²) was required to achieve the desired surface temperature within an 8- to 12-hour heatup period.

After the drying cycle is completed the bridge deck should be protected from rapid cooling to minimize cracking. The surface temperature drop during cooling should not exceed 30°F (17°C) per hour. The concrete is considered sufficiently cool when the temperature as measured at the 1-inch (25-mm) depth is not greater than 100°F (38°C).

4. After cooling a layer of dry sand should be placed on the concrete to be impregnated. The sand layer should be 1/4 to 3/8 inch (6.4 to 9.5 mm) thick. The sand gradation is not critical provided that particle sizes greater than those retained on a No. 16 sieve or less than those passing a No. 100 sieve are not present in quantities greater than 2 percent. The sand should be uniformly blended and clean, free of clay, silt, or organic matter.

A sand gradation that has worked well in the field is:

<u>Screen size</u>	<u>Percent passing</u>
# 16	98
# 30	64
# 50	32
# 100	0.02

5. The monomer system used for impregnation should be composed of 90 to 97 percent MMA and 10 to 3 percent TMPTMA by weight. These components may be premixed, as convenient, prior to use. Just before application to the bridge deck a polymerization catalyst, AMVN, should be added to the monomer at a rate of 1/2 to 1 percent by weight and thoroughly mixed. Monomer temperature at the time of catalyst addition should not exceed 85°F (29.5°C).

After mixing, the catalyzed monomer should be uniformly applied to the concrete at a total rate of 1.2 to 1.3 lb/ft² (5.86 to 6.35 kg/m²). Application should begin at the superelevated side of the deck, if any, and proceed to the lower side. The monomer may be applied in two applications if necessary to prevent excess monomer from flowing off the deck. However, the second application should follow the first as soon as the sand will accept the additional monomer (normally less than 2 hours). As soon as monomer has been applied to any portion of the deck, the monomer-saturated area should be covered with polyethylene sheeting to reduce evaporation and fire hazard. The monomer should soak into the concrete for a minimum of 8 hours. In no event, however, should polymerization heat be applied to the deck if there is excess monomer in the sand. A simple test to determine the presence of excess monomer is to take a handful of sand and squeeze it. If monomer can be squeezed from the sand an excess is present and either the soak cycle should be continued or the excess monomer removed or evaporated from the affected area. (Gloves should be worn when squeezing the sand).

6. After the monomer has impregnated the concrete it should be polymerized in situ by heating the deck until a uniform temperature of 160°F (71°C) is obtained on the surface of the concrete. This temperature should then be maintained in the range of 160° to 180°F (71° to 82°C) for a minimum of 6 hours to complete polymerization.

6.2 Safety Provisions

Adequate provisions must be taken during the treatment process for a safe operation. There are numerous safety regulations, such as the Occupational Safety and Health Act of 1970 as amended, that are generally applicable to the treatment process and these should be known by prospective users of this procedure. Those provisions and regulations will not be detailed. The requirements that must be followed to reduce the hazards associated with using the recommended monomer and catalyst will be described.

The surface impregnation treatment must be conducted under the supervision of technically competent personnel trained in the handling and use of monomers and catalysts. All workmen must be instructed in the proper handling and use of these materials and in emergency practices in the event of an accident.

The manufacturers and suppliers of MMA, TMPTMA, and the polymerization catalyst, AMVN, provide excellent literature detailing safe handling and use provisions for these materials. This literature should be obtained and studied prior to beginning a bridge treatment project. In no event should this report or any section of it be used as a substitute for the manufacturer's literature.

The monomer, MMA, a thin liquid, must be handled and used in accordance with established procedures to insure operational safety. Three potential hazards may be encountered with MMA: flammability, toxicity, and the possibility of accidental bulk polymerization. Liquid MMA is highly flammable (Class I liquid) and can evaporate to form explosive mixtures with air. It, thus, must be protected from excessive heat, sparks, and other sources of ignition. Storage facilities must be properly ventilated. Monomer should be kept cool, out of direct sunlight, and away from open flames and oxidizing agents. Electrical appliances, lighting, and power sources must be in compliance with explosionproof requirements of the National Electrical Code. Obviously, smoking must not be permitted in and around storage facilities and other areas where MMA is being used. As a practical rule with respect to flammability, the precautions required for the safe use of gasoline will result in the safe use of MMA. However, the procedures should also conform with local ordinances and the requirements of underwriters and insurance companies.

MMA, if taken internally, is toxic and the manufacturers warn that skin exposed to MMA may develop mild cases of dermatitis. The eyes are particularly sensitive to the monomer and as a minimum protection personnel using the monomer should wear protective glasses or goggles. Full face shields may be justified during the mixing operation. Rubberized gloves, aprons or coveralls, and boots will protect personnel from monomer exposure under most conditions experienced in bridge deck treatment. In any event, skin exposed to MMA should be promptly washed with soap and water. The vapors of MMA may be irritating to respiratory membranes and precautions should be taken to prevent long exposure to concentrated vapors. It should be noted, however, that MMA, even in very low concentrations, has a strong characteristic odor. The presence of this odor, even though quite strong, does not necessarily indicate excessive vapor concentrations.

Accidental polymerization of bulk quantities of monomer must be prevented. The most probable cause of such an accident is storage of catalyzed monomer or catalyst addition to monomer at elevated temperature. In some instances impurities from dirty storage or mixing equipment may cause accidental polymerization.

Accidental polymerization of bulk monomer creates two hazards. First, the polymerization reaction is exothermic, i.e., generates heat. This heat may cause bulk polymerization in adjacent monomer containers, damage other equipment, or cause personnel burns. Second, as the temperature reaches the boiling point of the polymerizing monomer, approximately 212°F (100°C), the monomer vapor pressure increases, and in closed containers the resulting pressure increase may cause the container to rupture explosively. This sudden release of large quantities of monomer vapor and hot, polymerizing monomer creates critical flammability and personnel hazards. Because of these bulk polymerization hazards, storage of monomer containing catalyst is not recommended.

Monomer spills present fire and environmental hazards. Precautions should be taken so that in the event of an accidental spill the monomer will be safely contained. Spilled monomer may be absorbed in a material such as vermiculite, which should then be handled with nonsparking tools for disposal. Vapors from a large spill in a confined area may also present health hazards (see ACGIH publication "Threshold Limit Vapors for Chemical Substances and Physical Agents in the Workroom Environment"). In this event, removal of the monomer may require a trained crew wearing supplied air respirators.

Disposal of waste or excess monomer, particularly if catalyzed, is troublesome at best. For this reason every reasonable and practical effort should be made to prevent spills, monomer contamination or catalyst addition to more monomer than an impregnation project requires. In the event that monomer disposal is necessary, two general procedures are available - incineration and bulk polymerization. Incineration of excess monomer or monomer-saturated material is self-explanatory. However, compliance with local, state, and Federal regulations applicable to the incineration process is mandatory. Bulk polymerization of MMA or MMA-TMPTMA mixtures is accomplished by adding a polymerization catalyst and a chemical promoter or heat to small quantities of the monomer in open top containers and allowing the monomer to polymerize. The resulting inert polymer may then be disposed of in conventional landfill facilities. Special precautions, however, are required to perform this disposal technique safely. For this reason the assistance of a technical advisor experienced in the bulk polymerization disposal process should be obtained to train personnel contemplating using this waste disposal technique.

Monomer disposal by dumping, landfill, or transfer to a sewage system is not acceptable.

The liquid monomer, TMPTMA, although combustible is not flammable and for all practical purposes is not considered toxic. The major hazard lies in its propensity to bulk polymerize. For this reason it is supplied from the manufacturer containing 200 parts per million hydroquinone (HQ) as inhibitor (MMA normally is supplied with 25 parts per million HQ). This stabilizes the monomer under normal conditions and if the storage and use provisions for MMA are met there should be no problem with TMPTMA or with MMA-TMPTMA mixtures over the short storage periods normally expected in the field. For storage periods longer than 3 months the monomer manufacturers should be contacted to obtain appropriate storage information.

The polymerization catalyst, AMVN, is flammable, toxic, and temperature sensitive. It is a dry powder and must be stored and used in accordance with the recommendations of the supplier. These recommendations generally include storage in explosionproof facilities at temperatures lower than 50°F (10°C). Personnel working with this material should be required to wear protective gloves and safety glasses. In no event should this dry powdered material be used in a manner that will create air suspended dust that is toxic and explosive.

7. CRACKS

In the Field Tests section of this report the occurrence of circular pattern cracking on the decks of the Denver, Idaho, and California bridges was discussed. These cracks were 2-1/2 to 3 inches (63.5 to 76.2 mm) deep, of hairline width, and formed circular patterns on the deck surfaces roughly 4 to 12 inches (102 to 305 mm) in diameter and extended over the entire treated areas of the decks.

7.1 Investigations to Determine the Cause of the Cracks

Microscopic and petrographic examination of cores from the Denver bridge eliminated adverse cement-aggregate or monomer/polymer-concrete reactions as potential causes of the cracking. The concrete in this bridge deck was relatively young (2 weeks old at beginning of the drying cycle). It was suspected that the relatively low tensile strength of the concrete at this early age was insufficient to resist the expansion and contraction stresses of high-temperature drying. Laboratory tests were performed on concrete slabs during which 2-week-old concrete was dried under the extreme conditions of 400°F (204°C). During these tests the pattern cracking seen on the Denver bridge was not evident. The laboratory test slabs, however, were relatively small, 4 feet square (1.22 m square) and 3 feet by 6 feet (0.91 m by 1.83 m) and were tested in an unrestrained condition.

In preparation for the treatment of the Idaho bridge, specifications were written requiring the bridge contractor to water-cure the deck for 28 days prior to drying. This was done as specified and a close inspection of the deck prior to treatment indicated that the concrete was free of cracks. A second inspection of the deck immediately following the heating cycle revealed the existence of pattern cracking similar to that on the Denver bridge. Insufficient curing was thus eliminated as a cause of the cracking. The conclusion that monomer/polymer-concrete reaction was not a cause was confirmed as no monomer had been applied to the bridge at the time these cracks were seen.

Numerous discussions of the pattern cracking problem were held with engineers and scientists experienced in concrete technology. Ultimately, it was concluded that the circular pattern of the cracks was indicative of essentially uniform multidirectional stresses and that the cause of these stresses was drying shrinkage of the concrete during the drying cycle and possibly thermal contraction during cooling.

Based on these conclusions the solutions to the problem appeared to be to remove less water from the concrete during the drying cycle.

A new series of oven-drying tests was performed on 2-foot-square by 4-inch-thick (710-mm-square by 100-mm-thick) slabs using drying cycles of

24, 36, 48, and 60 hours duration after 250°F (121°C) concrete surface temperatures were reached. Oven temperatures were varied so that the concrete surface temperature was increased from ambient at a rate as near to 30°F (17°C) per hour as possible. Normally, 8 to 12 hours were required to reach the 250°F (121°C) concrete surface temperature. Thus, total heat cycle times varied from about 34 to 70 hours.

After these slabs were cooled, the top surface of each was impregnated with the 95-5 MMA-TMPTMA monomer system. Polymerization was accomplished at 160°F (71°C). The results of these tests are shown in table 15.

The slabs used in this test had been stored in indoor ambient air conditions for 6 to 8 months and at the beginning of heat application had relatively low moisture contents. A second series of tests was then performed using slabs that had been soaked in a water bath for 3 days. These results are shown in table 16.

The test results indicate that marginally acceptable polymer penetrations could be achieved with 24-hour drying times but that 36-hour drying times resulted in a slightly deeper penetration.

To determine the effects of shorter drying times on polymer penetration and the cracking problem under field conditions, a 10-foot by 32-foot (3.0-m by 9.7-m) section of a large concrete slab on grade was treated. This slab was constructed of unreinforced concrete, a nominal 6 inches (152 mm) thick. Drying was accomplished using a hot-air enclosure similar to that used on the Denver bridge and two gas-fired heaters. With this equipment 250°F (121°C) temperature on the concrete surface was obtained in 8 hours. This temperature was maintained for 36 hours to complete the drying cycle.

Once during the drying cycle (24 hours after 250°F (121°C) was reached - 32 hours total heat time) and again at the completion of the drying cycle, visual examinations of the heated concrete revealed no pattern cracking of the concrete surface. After drying, a 36-hour cooling period was required to bring the 2-inch (51-mm) depth concrete temperature down to approximately 100°F (38°C).

The concrete surface was then covered with a layer of dry sand. Half of the surface was impregnated with the 95-5 MMA-TMPTMA monomer system, the other half with an 85-15 MMA-TMPTMA monomer system. The higher concentration of TMPTMA was believed useful in filling cracks should they occur. Both monomer systems were catalyzed with 1/2 weight percent AMVN. Two monomer applications were made at a total rate of about 1.6 pounds monomer per square foot of surface area (7.8 Kg/m²). Total impregnation cycle time was 17 to 18 hours.

Table 15

SURFACE IMPREGNATION DRYING TIMES
2-FOOT X 2-FOOT X 4-INCH SLABS (AIR DRIED CONCRETE)

No.	Time at 250°F hours	Polymer depth inches
130	24	15/16 - 1-1/4
131	36	1-1/8 - 1-3/8
132	48	1-1/2 - 1-5/8
133	60	1-3/4 - 1-7/8

Table 16

SURFACE IMPREGNATION DRYING TIMES
2-FOOT X 2-FOOT X 4-INCH SLABS (WATER-SOAKED CONCRETE)

No.	Time at 250°F hours	Polymer depth inches
134	24	3/4 - 1
136	24	3/4 - 1
135	36	1 - 1-3/16
137	36	1 - 1-3/16
138	36	1-1/4 - 1-5/8

At the completion of the impregnation cycle the enclosure and heat system were reinstalled and polymerization accomplished by heating the concrete to 180°F (82°C) for 18 hours. Cores taken from the treated area exhibited polymer penetrations of 3/4 to 1-1/2 inches (19 to 38 mm) with no significant difference between the two monomer systems. Some 50 percent of the surface was covered by an overlay formed by sand and the monomer containing 15 percent TMPTMA; however, the circular pattern cracking, which had been observed on the Denver and Idaho bridges, could not be detected in the clear areas of the slab.

A 24-hour drying cycle at 250°F (121°C) was selected to begin the surface impregnation treatment of the California bridge.

As discussed in section 5 of this report, the 24-hour drying cycle (36-hour total heat application) and a subsequent 12-hour drying cycle (24-hour total heat application) gave acceptable polymer penetrations on the California bridge, but both resulted in the formation of surface cracking as experienced on the Denver and Idaho bridges.

Following the California test, a review of the conditions during the drying cycle was made. At the beginning of drying the concrete surface is normally at a temperature of 50° to 80°F (10° to 27°C). After the heaters are turned on the concrete is exposed to a relatively high velocity blast of hot air which very quickly reaches a temperature in excess of 300°F (149°C). The concrete begins to absorb this heat and its temperature increases. One theory is that during the early stages of the drying cycle the combination of high-velocity airflow and high air temperature causes a very rapid moisture loss from the concrete just at the air-concrete interface. It is believed that at this point excessive drying shrinkage stresses are created on the surface of the concrete resulting in surface cracks which subsequently propagate downward to the upper level of the reinforcing steel.

An alternate theory is that large thermal stresses are generated when the heaters are turned off and the concrete begins to cool. The contraction upon cooling, combined with the effects of drying shrinkage, would produce cracking, or if the cracking were already present from the initial heating, the cracks would be deepened.

In either event, i.e., the initial rapid surface drying or the subsequent cooling or a combination of both, the problem should be alleviated by providing a buffer zone on the concrete surface.

Oral communications with researchers at Lehigh University who are working on a technique of impregnating concrete to a depth of 4 inches (102 mm) indicate that a layer of sand placed on the concrete surface prior to drying is effective in eliminating the surface spalling associated with drying at temperatures of 400°F (203°C) and higher. Similarly,

a portion of a bridge deck was surface impregnated by the University of Texas at Austin. During this treatment a layer of undried sand was placed on the deck prior to drying, both to dry the sand before impregnation and to provide a more uniform heat distribution to the concrete. During the drying cycle, concrete surface temperatures greater than 300°F (149°C) were obtained. It is reported that cracks of the type seen on the Denver, Idaho, and California bridges did not occur.7/

These efforts appear to support the use of a sand layer during drying to prevent the formation of surface cracking. It should be pointed out, however, that the surface areas treated with this technique have been relatively small, less than 350 square feet (32.5 m²) and it is not certain that these smaller areas would have experienced the surface cracking without the sand layer. In any event, a full-scale bridge treatment is scheduled for the summer of 1976. During this treatment, a sand layer will be applied to the concrete deck prior to the drying cycle to evaluate its effectiveness in preventing pattern cracking.

7.2 Tests to Repair the Cracked Deck Surfaces

As previously stated, the pattern cracks that developed in the three bridges are hairline width and extend 2-1/2 to 3 inches (63.5 to 76.2 mm) into the concrete. These cracks are not a structural threat to the bridges. The cracks in the Denver bridge were closely observed for a period of almost 2 years. With exposure to traffic the cracks became more apparent and it was feared that they were becoming wider and possibly extending deeper into the deck. During the spring and early summer of 1976 numerous cores were taken through the cracked areas. Close inspection of these cores revealed, however, that the depth and width of the cracks had not changed and that what appeared to be increased crack width was abrasion-caused erosion of the concrete at the crack intersection with the riding surface of the deck.

Although the cracks are not structurally detrimental, it is believed that they may be sufficiently open to permit water and chloride intrusion into the concrete.

A series of tests was begun to develop a monomer system and application technique to fill or seal the cracks with polymer. These tests have not been completed and only partial test results are available.

A monomer system for this application must meet a unique set of requirements. The viscosity must be low to permit penetration into the very fine cracks. Once in the cracks the monomer must remain there during polymerization to provide a solid barrier to moisture and chloride intrusion. No single monomer appeared to meet these requirements and a screening test was performed with comonomer systems of MMA, TMPTMA,

styrene (S), butylacrylate (BA), and vinyl ester (VE), and rubber modified vinyl ester (RVE). In all, over 60 monomer systems were screened for crack repair applications. Seven of these systems were selected for trial test application on the Denver bridge as listed in table 17. The relatively short soak times required to penetrate the cracks permitted the use of promoter-catalyst polymerization systems.

Two types of application techniques were considered: (1) A simple gravity soaking and (2) pressure injection by either individual crack injection methods or by a pressure mat. As the pressure injection methods would be very expensive for treating a large expanse of fine cracks, a decision was made to first extensively investigate the possibilities of using the much simpler gravity soaking technique.

Trial tests were performed on the bridge on three different days after there had been at least four rainless days prior to test.

For the first trial two separate 4-foot-square (1.2-m-square) areas were marked on the bridge deck. Each area was divided into four equal sections. One area was then dried with an infrared heater by heating the concrete surface to 300°F (149°C) and maintaining that temperature for 2 hours. The second area was not dried. After drying the concrete was cooled to 115°F (46°C) at the 1-inch (25-mm) depth before monomer application. Calking compound was placed around the perimeter of each of the eight test sections to create shallow reservoirs. The sections were numbered and monomer was applied directly to the concrete surface of each section as follows:

(Sections 1, 2, 3, and 4 were in the dried area. Sections 5, 6, 7, and 8 were in the undried area.)

<u>Section No.</u>	<u>Monomer system</u>
1 and 5	80 MMA - 18 - RVE - 1 S - 1 TMPTMA, 2 BP - 1 DMT
2 and 6	98 BA - 1 MMA - 1 TMPTMA, 1 BP - 1/2 DMT
3 and 7	95 MMA - 5 TMPTMA, 1 BP - 1 AMVN - 1/2 DMT
4 and 8	95 MMA - 5 TMPTMA, 2 AMVN

The monomer systems applied to sections 1, 2, 3, and 5, 6, 7 contained a chemical promoter and began to gel in about 15 minutes. All cured within 45 minutes. The monomer system applied to sections 4 and 8 was polymerized by heating the concrete to 160°F (71°C) with the infrared

Table 17

CRACK REPAIR MONOMER SYSTEMS
USED ON DENVER BRIDGE

Monomer % by weight	Catalyst % by monomer weight	Promoter % by monomer weight
60 - S 40 - TMPTMA	1 - BP	1/2 - DMT
95 - MMA 5 - TMPTMA	2 - AMVN	None
95 - MMA 5 - TMPTMA	1 - BP 1 - AMVN	1/2 - DMT
90 - MMA 9 - RVE 1 - TMPTMA	1-1/2 - BP	3/4 - DMT
80 - MMA 18 - RVE 1 - S 1 - TMPTMA	2 - BP	1 - DMT
90 - MMA 5 - RVE 5 - VE	1 - BP	3/4 - DMT
98 - BA 1 - MMA 1 - TMPTMA	1 - BP	1/2 - DMT

MMA = Methyl methacrylate
 S = Styrene
 RVE = Rubber modified vinyl ester
 VE = Vinyl ester
 BA = Butyl acrylate
 TMPTMA = Trimethylolpropane trimethacrylate
 BP = Benzoyl peroxide
 DMT = Dimethyl para toluidine

heater after a 1/2-hour soak time. On the day following monomer application test cores were taken from each of the eight sections and inspected microscopically for polymer penetration. Each of the monomer systems had penetrated the cracks and polymer was found throughout the crack interfaces. Only the promoted 95-5 MMA-TMPTMA system (sections 3 and 7) appeared to have filled the cracks to a depth greater than 1/2 inch (13 mm). There was no apparent difference in the results obtained on the dried and undried areas. A second test was performed with the promoted 95-5 MMA-TMPTMA monomer system containing 1 percent BP and 1 percent AMVN but the promoter concentration was reduced to 1/4 percent. The application technique was changed to allow a longer soak time before polymerization. Two 3-foot-square (910-mm-square) areas were prepared on the bridge deck surface. One was dried and cooled as in the first test. Calking compound was applied around the perimeter of each area to form shallow reservoirs to contain the monomer. Just prior to monomer application a 1/4-inch (6.4-mm) layer of dry sand was spread over the areas. Monomer was applied to the sand at a rate of 1/2 pound per square foot of surface area (2.4 Kg/m^2). The areas were closely observed and just after the monomer began to gel (approximately 1-1/4 hours after the application) the polymerizing monomer-sand composite was removed from the areas with a shovel. Test cores were taken from the areas on the following day and examined microscopically.

The top surface of the cracks appeared sealed with polymer which filled the cracks to a depth of approximately 1/2 inch (13 mm). There was polymer in the cracks to the bottom but the cracks were not filled with polymer below 1/2 inch (13 mm). Dark zones of impregnated concrete approximately 1/8 inch (3.2 mm) wide on either side of the crack planes were apparent in the zone below the surface impregnated concrete. There was no difference in the results on the dried or undried concrete. From these results, it was concluded that the low viscosity MMA-TMPTMA monomer system was successfully penetrating the cracks but was being lost through diffusion into the concrete before polymerization occurred.

In a third field test three monomer systems with slightly greater viscosities were tested. Three adjacent 3-foot-square (910-mm-square) areas were prepared with calking compound and sand as in the second test. None of the areas were dried. The first area was treated with a monomer system composed of 60-40 S-TMPTMA containing 1 percent BP and 1/2 percent DMT. The second area received a monomer system composed of 90 percent MMA, 9 percent RVE, and 1 percent TMPTMA and containing 1-1/2 percent BP and 3/4 percent DMT. The third area was treated with a 90 percent MMA, 5 percent RVE, 5 percent VE monomer system containing 1 percent BP and 3/4 percent DMT. The application rate at each of the three areas was 1/2 pound per square foot (2.4 Kg/m^3). The 60-40 S-TMPTMA system began to gel in 50 minutes while the two MMA systems began to gel in about 70 minutes. The polymerizing monomer-sand composites were removed from the concrete just after they began to gel. As in previous tests, cores were taken from the treated areas and examined for polymer penetration.

The 60-40 S-TMPTMA system sealed the top surface of the cracks and appeared to fill the cracks to a depth of about 3/4 inch (19 mm). Below this depth the cracks contained polymer but were not filled. The dark zone associated with monomer diffusion was not evident in concrete treated with this system. Cores taken from the area treated with the 90-9-1 MMA-RVE-TMPTMA system contained cracks that were only partially sealed on the top surface and to a depth of 3/4 inch (19 mm). The monomer had diffused into the concrete below the zone of surface impregnation. The 90-5-5 MMA-RVE-VE monomer system solidly sealed the top surface of the cracks and filled one crack to the bottom. A larger adjacent crack was only filled to a depth of 3/4 inch (19 mm). This monomer system diffused into the concrete below the zone of surface impregnation resulting in a 1/4-inch (6.4-mm) wide zone of impregnated concrete along the crack plane.

As previously stated, this test series is continuing. The results obtained to date indicate that the crack repair technique is only partially satisfactory. The benefits of monomer impregnation around the crack zone have not been evaluated. It is possible, however, that such impregnation will prevent chloride intrusion into the unprotected concrete. In any event, emphasis in future studies will center on filling the cracks with polymer.

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APPENDIX A

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Comparison of Skid Resistance Between a Polymerized - Monomer Treated Portland Cement Concrete and Untreated Portland Cement Concrete as Affected by Laboratory Polishing of Three Different Macro Textured Wearing Surfaces.

by

Eugene J. Morawski

Work Performed at the Central Laboratory of
Maryland State Highway Administration at the
request of:

United States Department of Interior
Bureau of Reclamation
Engineering and Research Center
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April 1975

CIRCULAR TEST TRACK EVALUATION OF POLYMER-IMPREGNATED CONCRETE

During October of 1974, specimens were received from the Bureau of Reclamation, Denver, Colorado, to be tested in accordance with instructions accompanying letter of June 28, 1974.

The first set of 16 specimens (series A) was placed into Maryland Test Track in accordance with figure 1. After the tests were underway, Mr. Richard E. Hay visited the project and directed that 8 of the second set of 16 specimens (series B) be reground to a smooth surface texture. This was accomplished and, as a result, the three different surface textures were redesignated as:

- | | |
|---------------|---|
| 1. Normal | Very rough surface |
| 2. Semiground | As originally provided and termed
"Ground" by Colorado |
| 3. Ground | The new, smooth surface |

Data were gathered at pit stops as depicted in table 1, and in accordance with methods as follow:

1. B.P.T. - Measuring Surface Frictional Properties using the British Portable Tester, ASTM E 303. Readings were taken in the same two positions of each specimen and the results averaged.
2. Strain - This is a Maryland Experimental Method for expressing surface frictional properties by use of Strain Gages and a rotating wheel (Third Arm). This operation may be visualized by referring to appendix C, photographs 11 and 12. The independently powered third arm is revolving clockwise at peripheral speeds of 30 mi/h. Concurrently, the vertical shaft turns the entire three-wheel assembly clockwise at creep speed over the samples which are covered with 1/16 inch of water.

The resistance between the third arm revolving tire and sample surface is continually recorded through the strain gages, a transducer. This is recorded on a printout, photograph 12. At this time, numbers reported as strain should be regarded as simply relative measures of frictional resistance.

3. Wire Shadow - This is a Maryland Experimental Method of expressing surface texture as described in appendix A-1.

4. Sand Patch - This is a nationally "talked-about" method of expressing surface texture which we describe in appendix A-2.

The 16 specimens in series A represent 4 conditions as follows:

1. Control Normal
2. Impregnated Normal (Polymer-Impregnation)
3. Control Semiground
4. Impregnated Semiground

Figure 2 is included as an example depicting four individual specimens and their individual plots for all test parameters.

Figures 3, 4, 5, and 6 depict parameters of series A and B. Each line represents the average of four specimens.

Discussion

The Maryland test track is not a "wear" device. The three profile levels (normal, semiground, and ground) may be considered to have "worn" to those cross sections and then placed in test track to polish. Polishing in the test track simulates polish under actual automobile traffic. Analysis of data indicates in many instances differences between replicate samples exceeded differences among presumptively identical specimens, results were averaged, and somewhat of a trend toward decreased frictional resistance and texture with traffic was noted.

Conclusion

The Polymerized-Monomer treatment of the Portland Cement Concrete had no effect on the skid resistance or texture of the specimens at any of the three macro-texture levels examined.

The rate of polish was quite slow and was not affected by the monomer treatment.

FIGURE 1

Identification of Specimens and
Location in Test Track

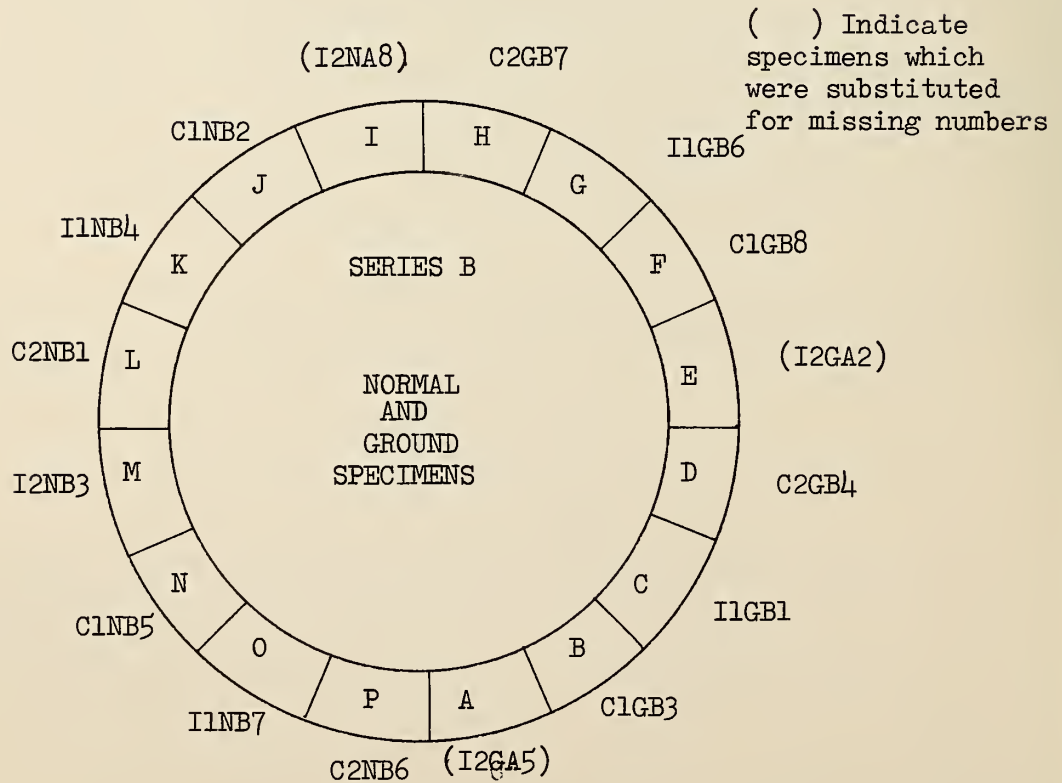
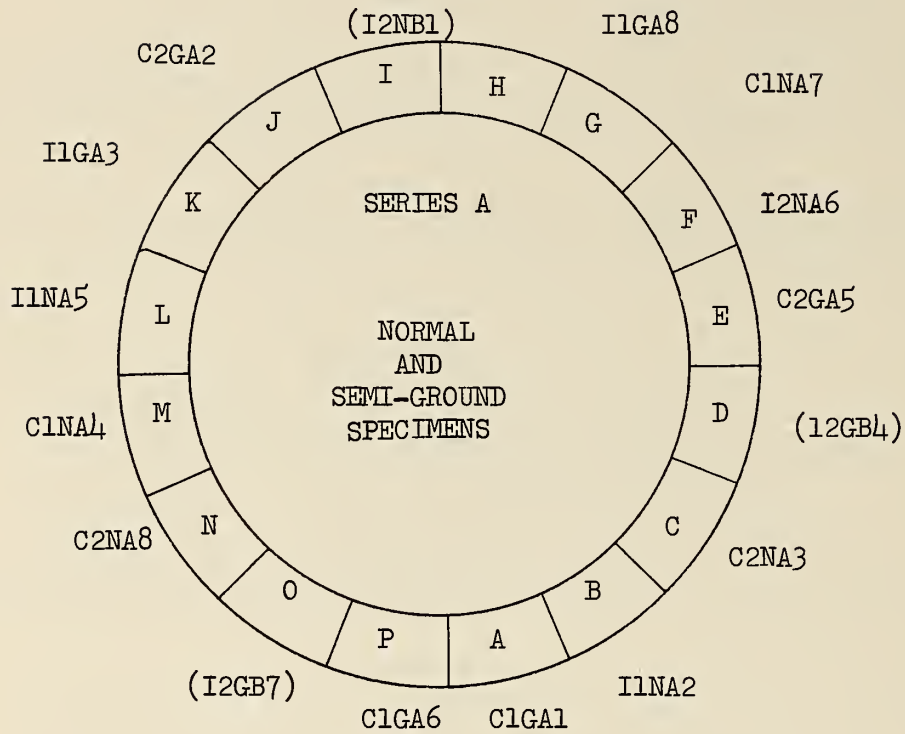


FIGURE 2

Example of Four Individual Specimens from Series A,
Control Semi-Ground

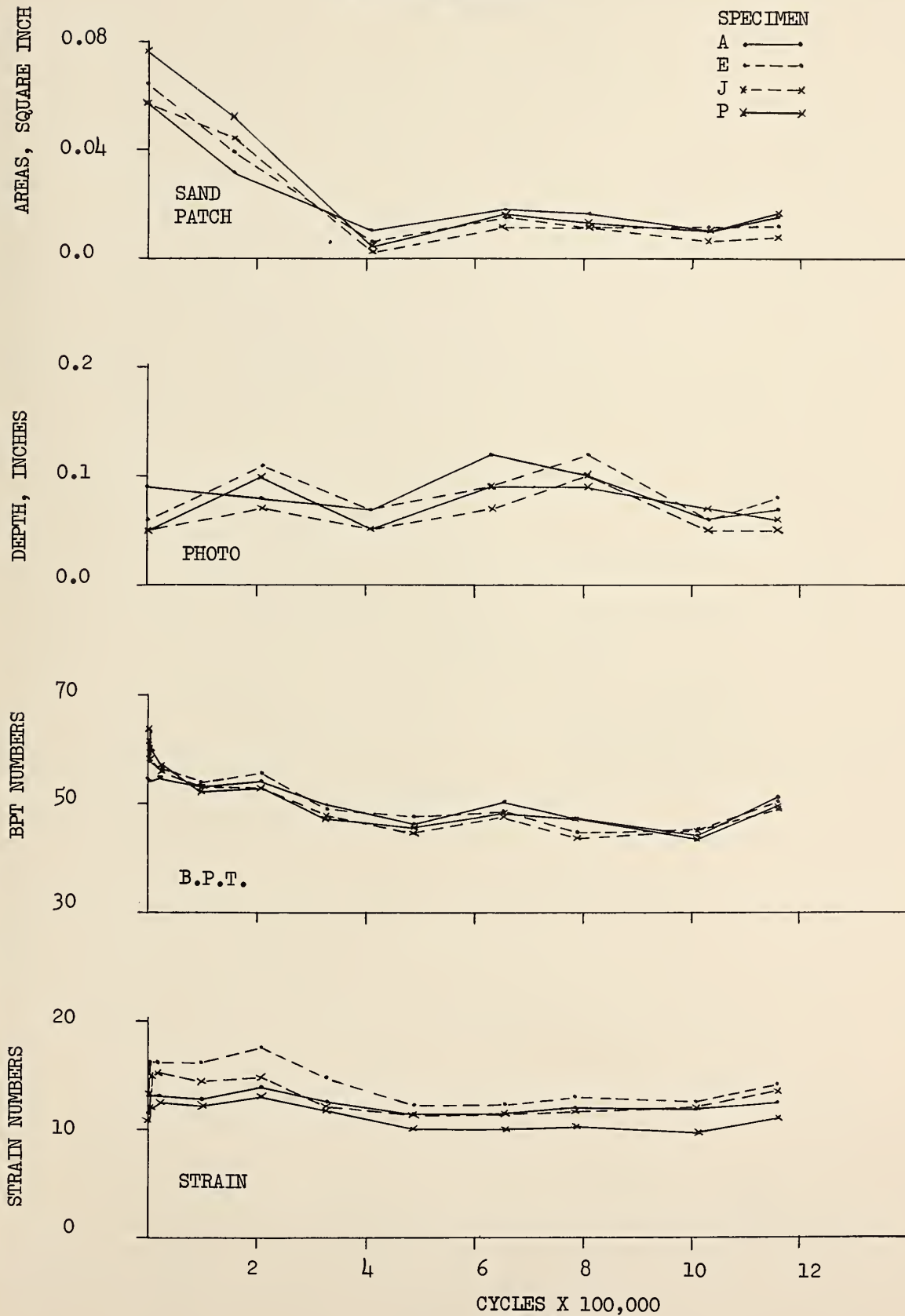


FIGURE 3
SERIES A

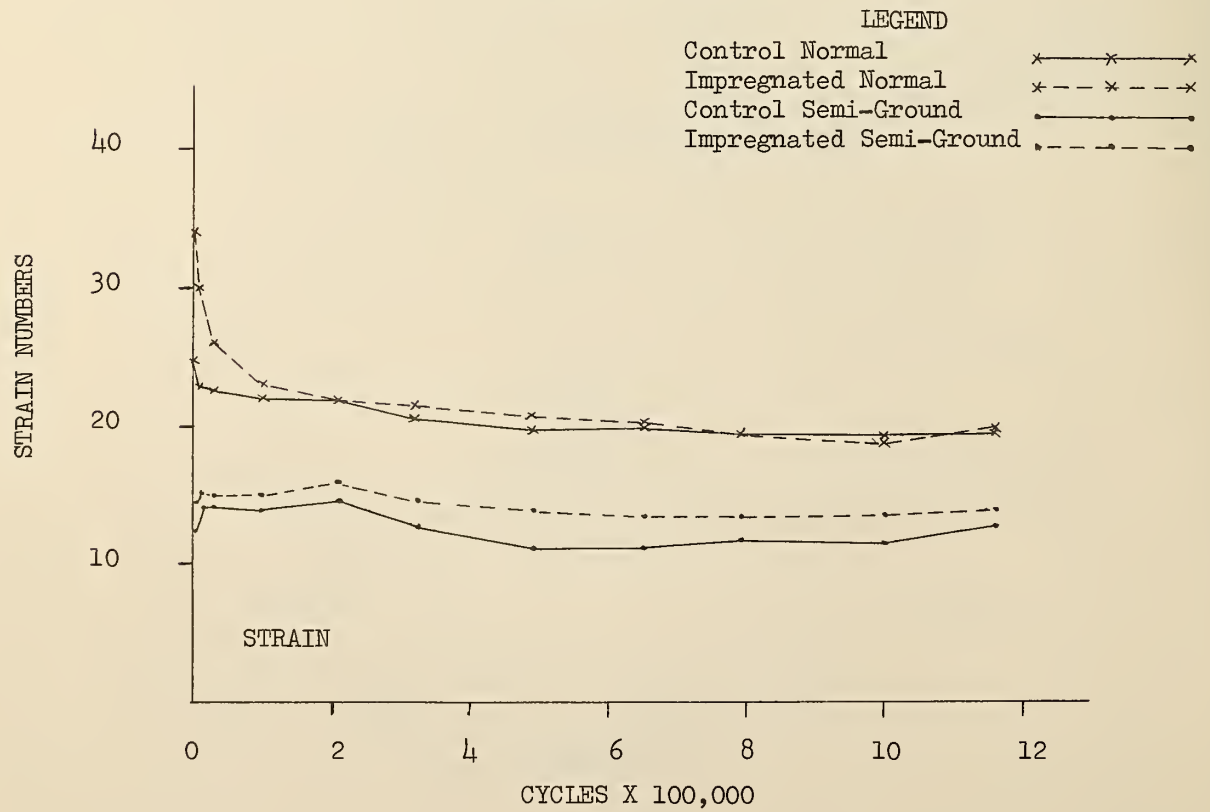
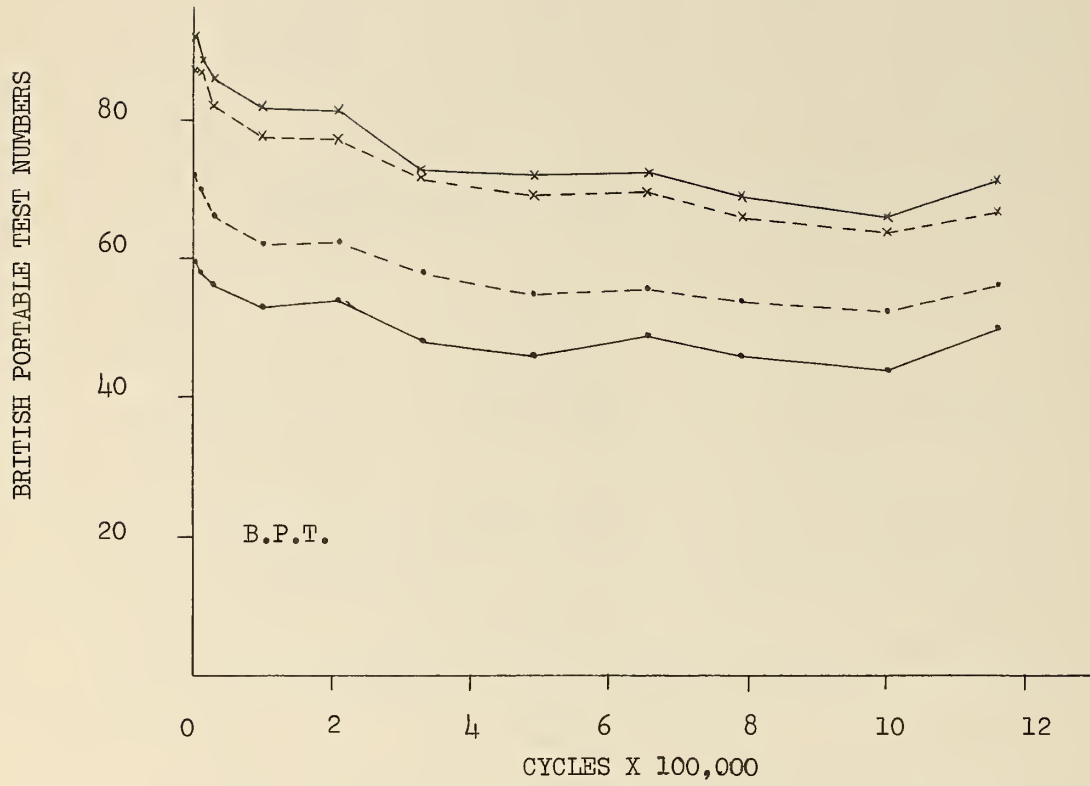


FIGURE 4
SERIES A

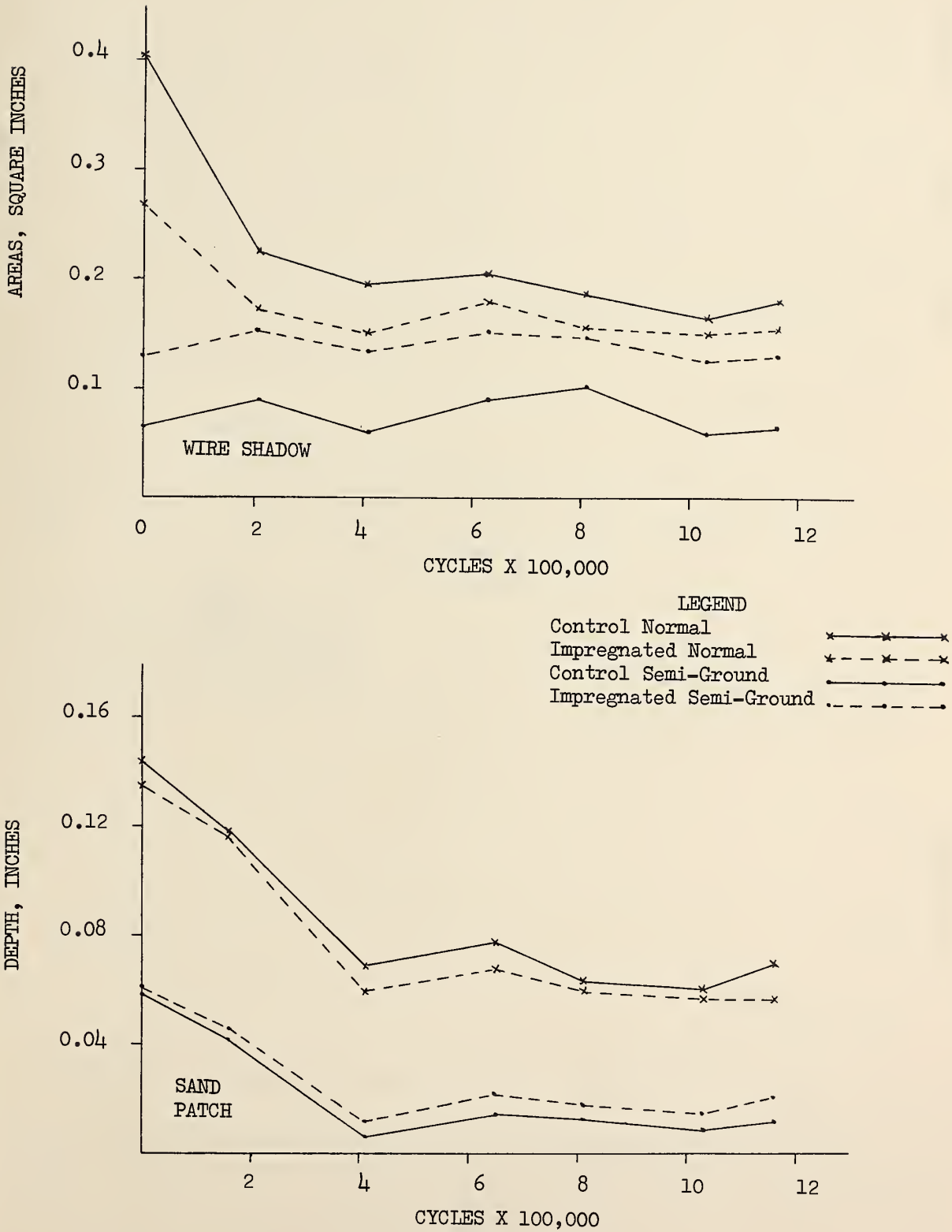


FIGURE 5
SERIES B

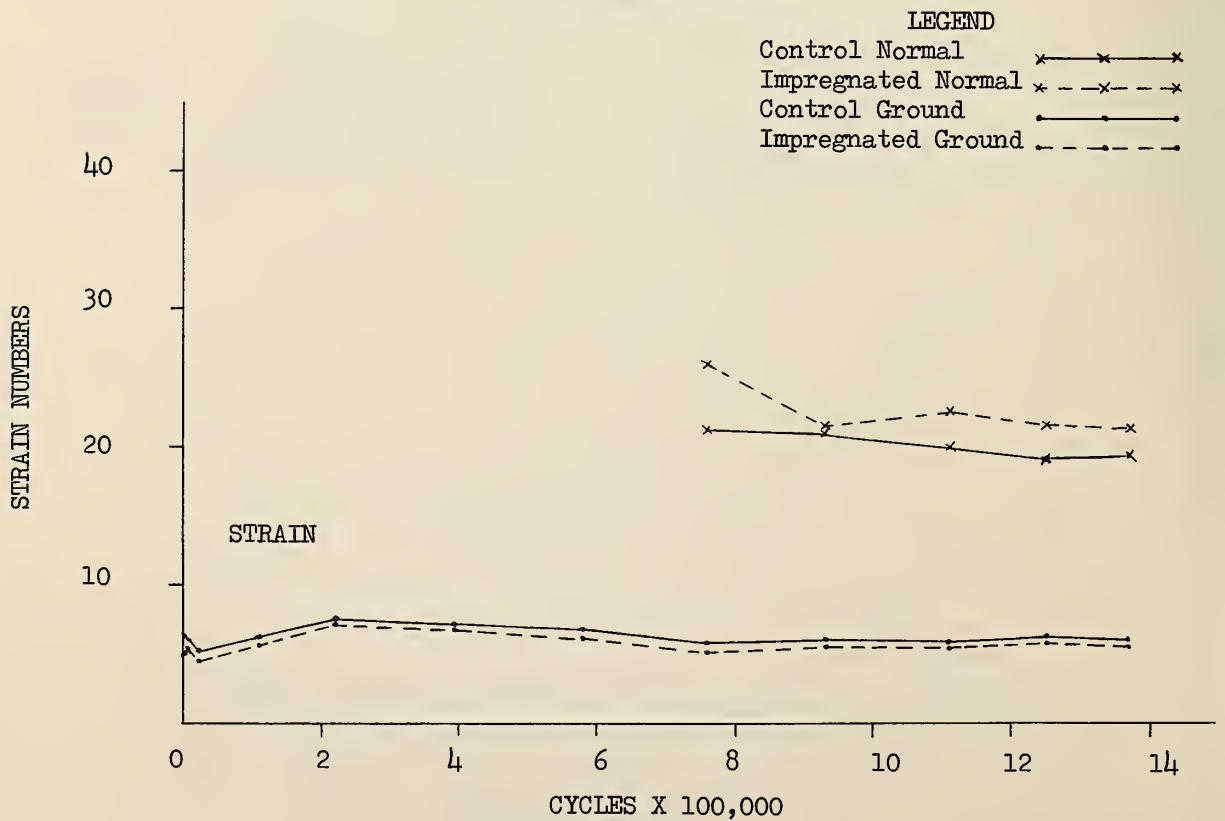
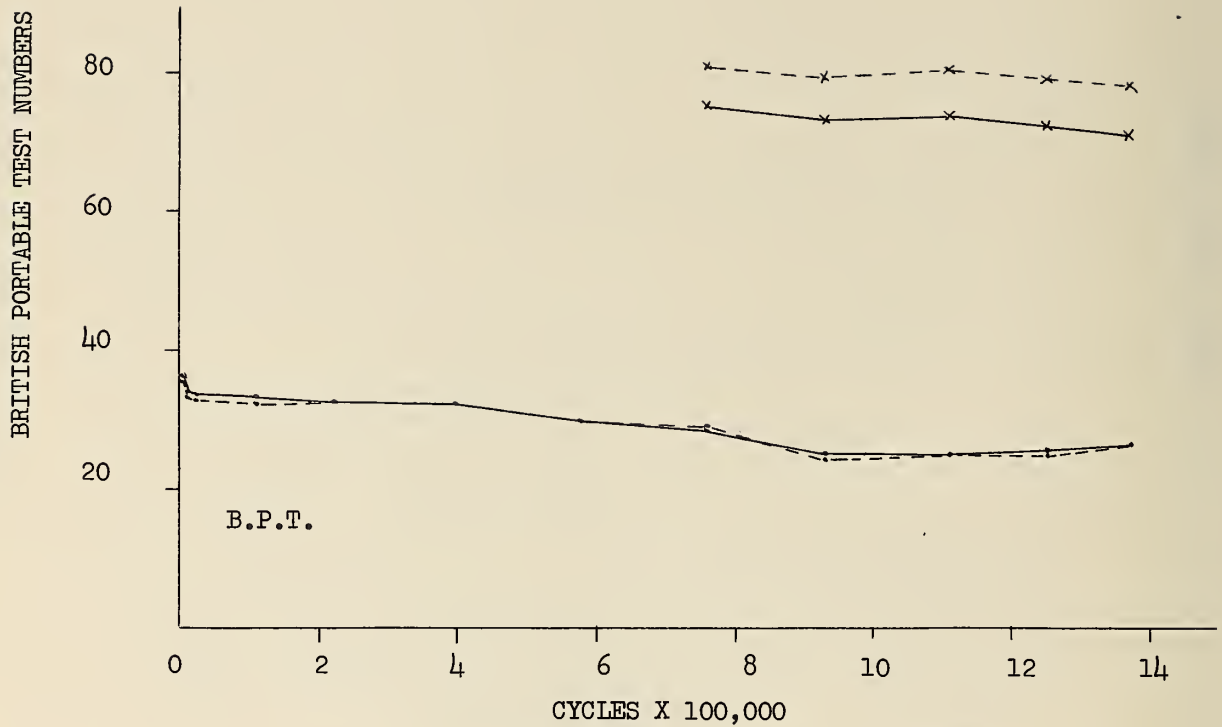


FIGURE 6
SERIES B

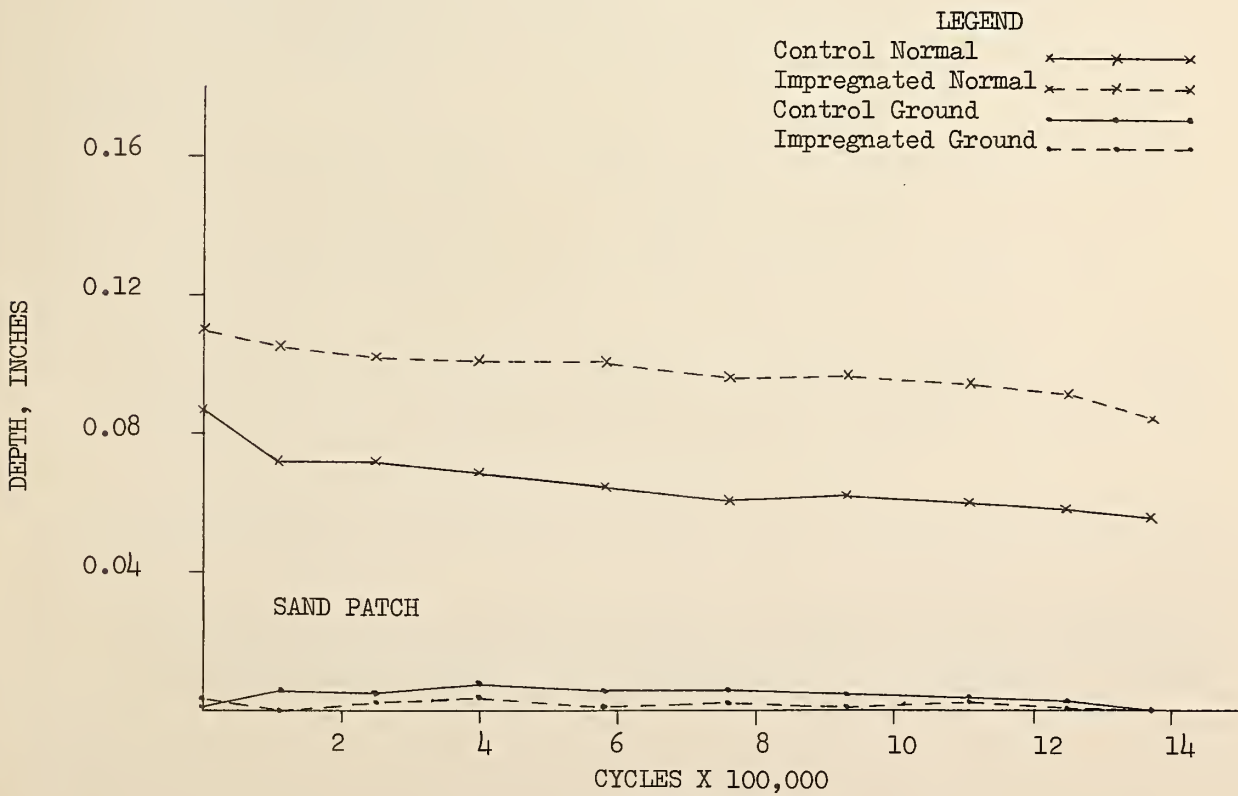
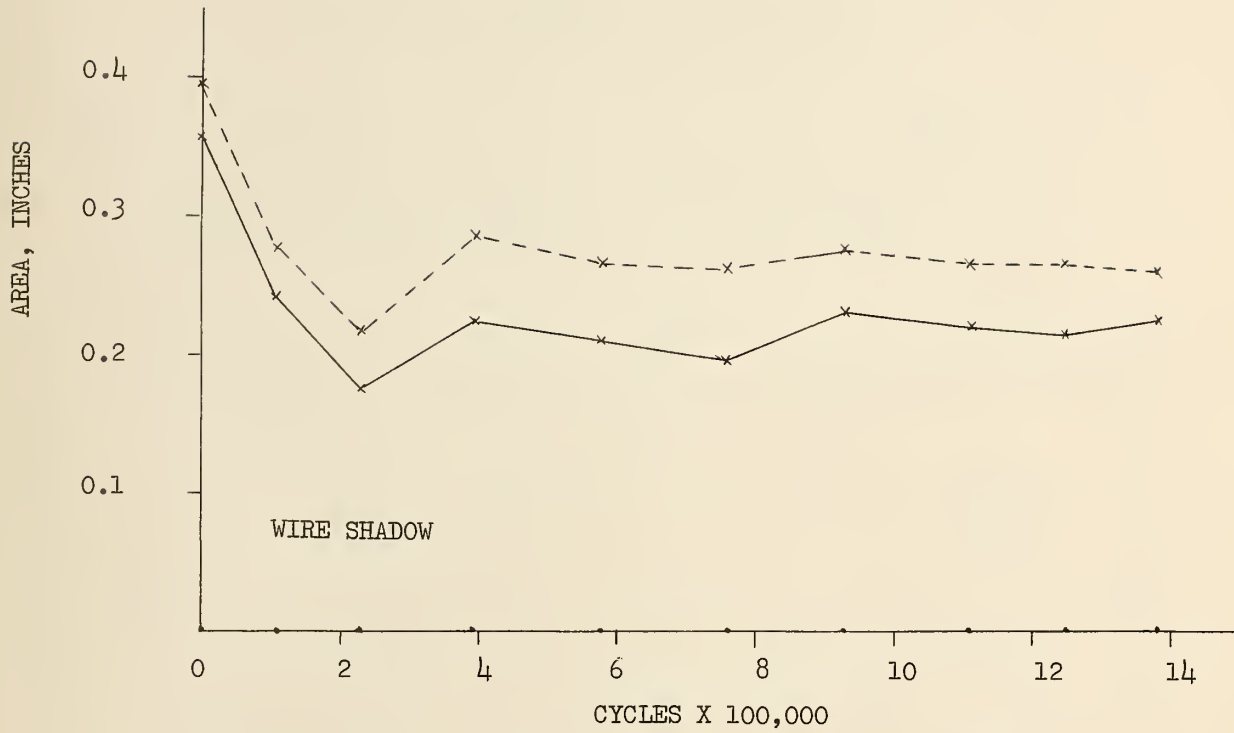


Table 1

THIS TABLE DEPICTS THE NUMBER OF REVOLUTIONS
AT WHICH VARIOUS DATA WERE GATHERED

Pit stop	Series A			Series B		
	Normal & semi-ground specimens			Normal specimens	Ground specimens	Normal & ground
	BPT 3rd arm	Photo	Sand patch	BPT 3rd arm	BPT 3rd arm	Photo sand patch
0		0	0			0
1	2,000			2,000	2,000	
2	6,000				6,000	
3	25,000				25,000	
4	102,470				111,000	111,094
4A			157,176			
5	210,500	210,520			224,383	224,379
6	330,000				396,600	396,600
6A		412,748	412,748			
7	493,100				580,220	580,217
7A		631,700				
8	655,400		655,401		763,550	
9	786,100				929,160	
9A		809,984	809,984			
10	1,009,000				1,107,000	
10A		1,031,663	1,031,663			
11	1,138,160	1,158,160	1,158,160		1,251,800	
12					1,374,000	

Note 1 - Original plan called for gathering all data at one pit stop in 1 day. This was unworkable. Rather than have "down time", a series A skid test was conducted 1 day, the track was run overnight, and texture measurements were conducted the next day.

Note 2 - B.P.T. and Strain data were not gathered until pit stop 8 in series b because the rough surface texture of specimens was damaging test equipment.

The project appeared ahead of schedule during series B. Therefore, the track was not run overnight and both skid and texture measurements were conducted at the same number of cycles.

Appendix A-1

EXPERIMENTAL MARYLAND TEST METHOD, 1975

Wire Shadow Method for Surface Texture

Scope: This method provides a means of determining changes in pavement surface texture by casting a shadow on a fixed angle, and photographing and measuring shadow area. The smaller the shadow area the smoother the surface. It is designed for use with samples in the Maryland Test Track, but may be modified for field use.

Equipment:

1. Grid Stand - Serves the dual purpose of supporting the camera 9 inches above the test surface and contains a wire grid system of known dimensions (figure 1)
2. Camera - Fitted with suitable lens for taking good quality closeup photographs
3. Light source - strobe
4. Light stand - Photographic tripod to hold strobe
5. Template - 1/8-inch aluminum sheet cut to shape of slightly smaller than track mold with center section 8-1/4 inch by 6-1/4 inch removed to allow for insertion of grid stand

Procedure:

1. Position light source on rigid support so that light strikes wire grid at a 45 degree angle. (figure 2)
2. Place the template atop the sample alining the scribe marks on its base and two sides with those on the track mold.
3. Place the grid stand, with camera, in template opening.
4. Focus camera, turn out room lights, take picture.
5. Repeat foregoing steps for remaining samples.

Photo Processing:

Develop the film and print each frame as an 8- by 10-inch enlargement. Care must be taken that all prints are enlarged to as nearly the same size each time.

Photo Evaluation:

Figure 3 depicts a portion of a typical photograph of the grid for evaluation.

1. Measure the width AB to the nearest 0.01 inch and record on work sheet. (figure 4)
2. Measure the distance from the grid wire to the closest point "c" (figure 3) of the shadow to the nearest 0.01 inch and record on worksheet. (figure 4)
3. Measure the distance from the grid wire to the best fit line (dotted line figure 3) through the shadow to the nearest 0.01 inch and record on worksheet. (figure 4)
4. Repeat steps 1 through 3 for the remaining four segments of each photograph.

Calculations:

Compute the test area as follows:

$$X = AB (D-C)$$

where:

X = the shadow area of the grid segment

AB = width of the grid segment

C = distance from the wire to the nearest point of the shadow

D = distance from the wire to the best fit line through the shadow

Report:

Report the average shadow area of the five grid segments, to the nearest 0.01 inch, as the test area for each sample section.

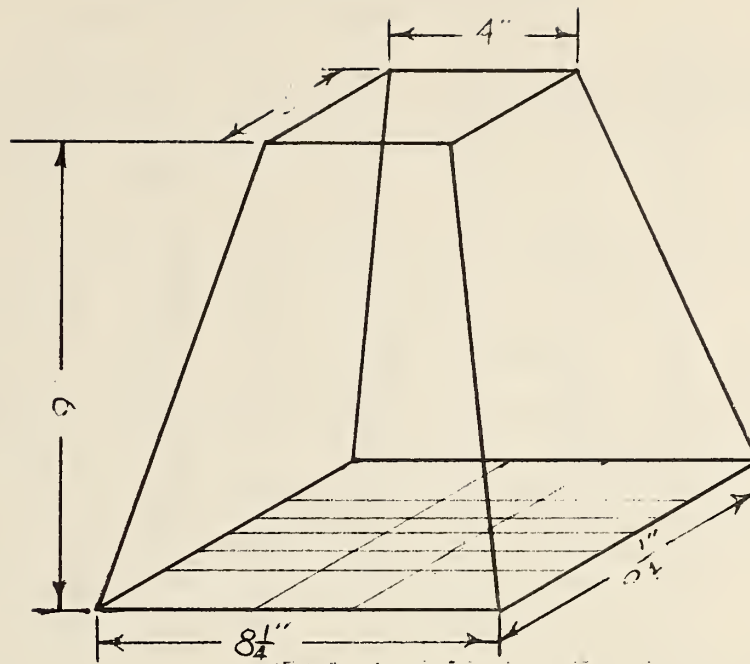


FIG. 1 - GRID - TANK

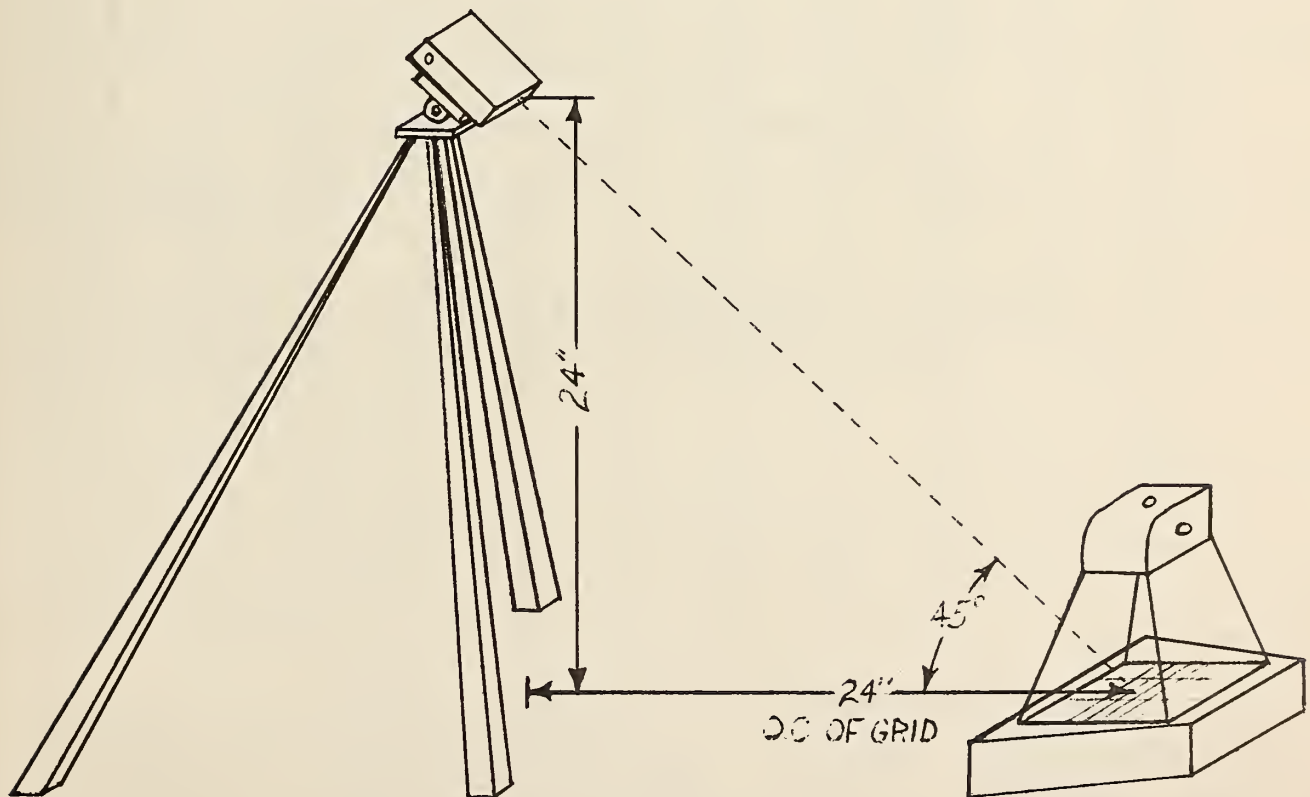
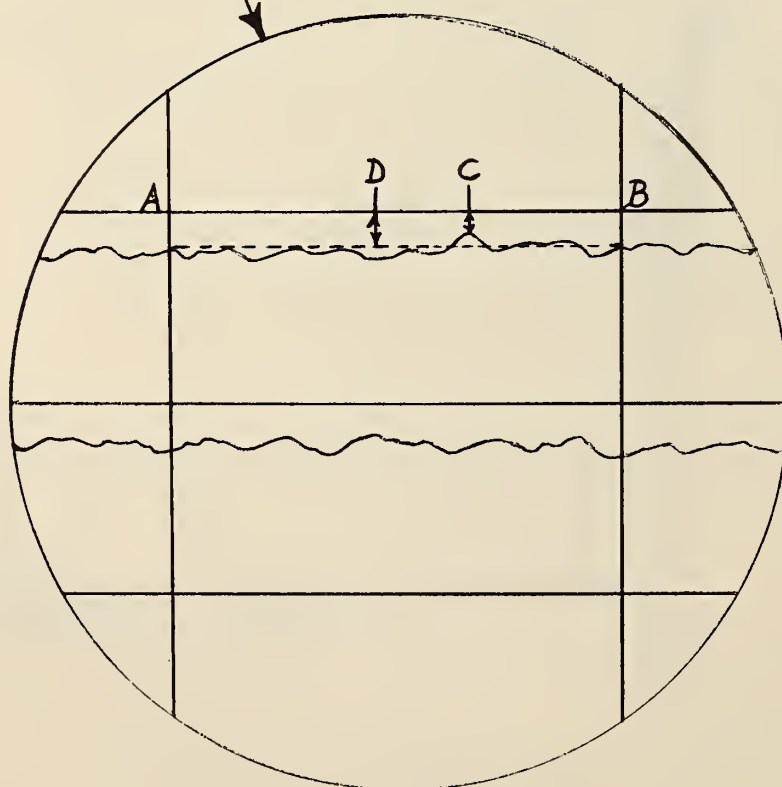
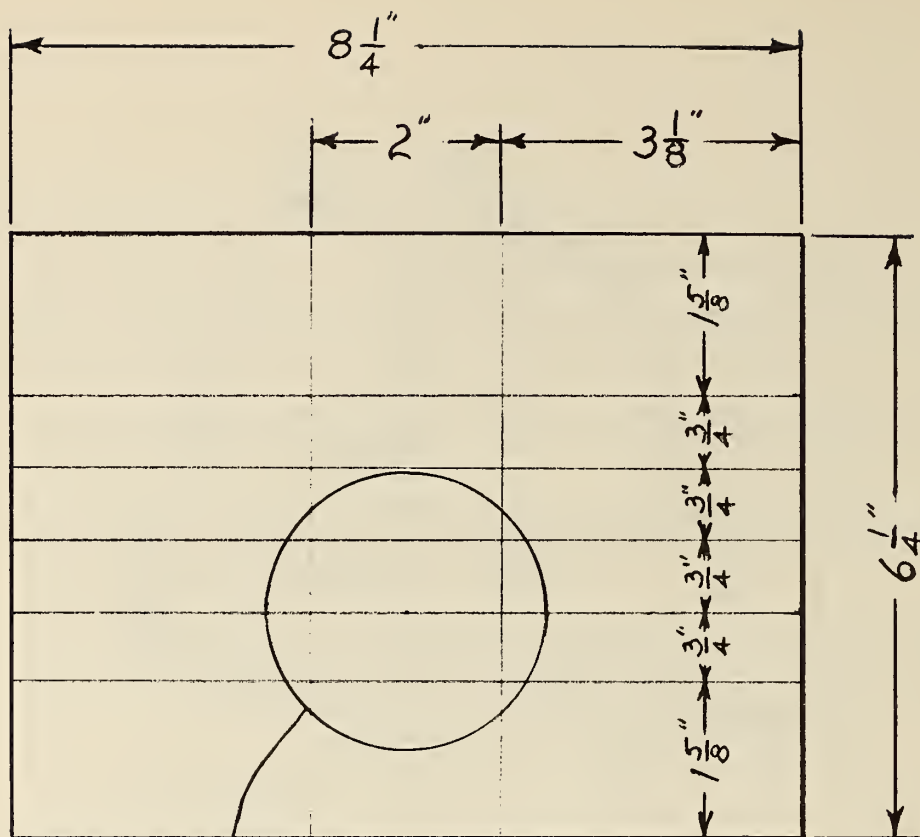


FIG. 2 - PROJECTED APPARATUS SET UP



~~~~~ SHADOW CAST  
 BY GRID  
 ---- BEST FIT  
 CENTER LINE

FIG-3

# AREAS OF PICTURE BY GERBER SCANNER

| SAMPLE LOCATION | AREA #1              | AREA #2              | AREA #3              | AREA #4              | AREA #5              | AVERAGE |
|-----------------|----------------------|----------------------|----------------------|----------------------|----------------------|---------|
| C16A1<br>A      | .26/3.28<br>.23 0.10 | .24/3.26<br>.22 0.07 | .24/3.25<br>.22 0.07 | .22/3.25<br>.21 0.03 | .19/3.28<br>.18 0.03 | 0.06    |
| I1NA2<br>B      | .34/3.31<br>.26 0.26 | .33/3.29<br>.26 0.23 | .32/3.26<br>.28 0.13 | .28/3.26<br>.21 0.23 | .26/3.29<br>.21 0.16 | 0.20    |
| C2NA3<br>C      | .40/3.29<br>.36 0.13 | .38/3.28<br>.34 0.13 | .35/3.26<br>.32 0.10 | .32/3.26<br>.28 0.13 | .29/3.27<br>.26 0.07 | 0.11    |
| I26B4<br>D      | .29/3.30<br>.24 0.17 | .28/3.26<br>.21 0.23 | .25/3.25<br>.19 0.20 | .24/3.26<br>.19 0.16 | .20/3.29<br>.15 0.16 | 0.18    |
| C26A5<br>E      | .29/3.29<br>.26 0.10 | .26/3.27<br>.24 0.07 | .27/3.26<br>.25 0.07 | .24/3.25<br>.23 0.03 | .20/3.27<br>.19 0.03 | 0.06    |
| I2NA6<br>F      | .33/3.30<br>.28 0.17 | .32/3.26<br>.28 0.13 | .30/3.25<br>.26 0.13 | .26/3.25<br>.23 0.10 | .25/3.28<br>.21 0.13 | 0.13    |
| C1NA7<br>G      | .34/3.30<br>.31 0.10 | .31/3.28<br>.27 0.13 | .30/3.26<br>.26 0.13 | .29/3.27<br>.24 0.16 | .25/3.27<br>.17 0.26 | 0.16    |
| I1GA8<br>H      | .32/3.31<br>.27 0.17 | .30/3.28<br>.24 0.20 | .26/3.26<br>.23 0.10 | .25/3.27<br>.23 0.07 | .23/3.28<br>.19 0.13 | 0.13    |
| I2NB1<br>I      | .33/3.32<br>.31 0.07 | .30/3.30<br>.29 0.03 | .29/3.28<br>.26 0.10 | .27/3.29<br>.25 0.07 | .25/3.29<br>.22 0.10 | 0.07    |
| C26A2<br>J      | .28/3.29<br>.27 0.03 | .26/3.27<br>.25 0.03 | .26/3.25<br>.24 0.07 | .25/3.26<br>.23 0.07 | .23/3.27<br>.22 0.03 | 0.05    |
| I1GA3<br>K      | .31/3.30<br>.27 0.13 | .30/3.28<br>.25 0.16 | .28/3.27<br>.25 0.10 | .26/3.28<br>.23 0.10 | .23/3.29<br>.20 0.10 | 0.12    |
| I1NA5<br>L      | .40/3.29<br>.32 0.26 | .35/3.27<br>.29 0.20 | .32/3.26<br>.24 0.26 | .30/3.25<br>.26 0.13 | .27/3.27<br>.23 0.13 | 0.20    |
| C1NA4<br>M      | .31/3.32<br>.24 0.23 | .29/3.31<br>.25 0.13 | .24/3.29<br>.17 0.23 | .22/3.27<br>.13 0.29 | .19/3.29<br>.14 0.16 | 0.21    |
| C2NA3<br>N      | .31/3.27<br>.27 0.13 | .30/3.26<br>.23 0.23 | .28/3.25<br>.22 0.20 | .25/3.25<br>.18 0.23 | .23/3.27<br>.18 0.16 | 0.19    |
| I26B7<br>O      | .28/3.29<br>.26 0.07 | .27/3.27<br>.25 0.07 | .27/3.26<br>.25 0.07 | .25/3.25<br>.23 0.07 | .23/3.27<br>.21 0.07 | 0.07    |
| C16A6<br>P      | .27/3.32<br>.24 0.10 | .25/3.29<br>.23 0.07 | .24/3.26<br>.22 0.07 | .24/3.26<br>.23 0.03 | .21/3.27<br>.19 0.07 | 0.07    |

## EXAMPLE

|          |      |
|----------|------|
| (D) (AB) |      |
| .26/3.28 | (X)  |
| (C)      | 0.10 |
| .23      |      |

AB = Width of Grid Segment  
 C = Distance from Wire to the  
 Nearest Point of the Shadow  
 D = Distance from the Wire to the  
 Best Fit Line Through the Shadow

FIGURE 4

OPERATOR J. Mitchell  
 DATE 12/12/74  
 REYS 1,031,663

TEXTURE MEASUREMENTS BY THE MODIFIED  
SAND PATCH METHOD

(As interpreted by Maryland for cooperative  
study for Bureau of Reclamation)

Scope

The technique involves determining the increase in the volume of sand required to fill a prescribed cavity when placed over a textured surface above the volume required to fill the same cavity when placed over a nontextured surface. The average texture depth is defined as the ratio of the increased volume of sand to the area of the patch.

Apparatus

1. A rectangular metal plate 6-3/16 by 4-5/8 by 1/4 inch thick with a 4-3/8- by 2-7/8-inch center hole, attached to the bottom of a 1/8-inch-thick striker plate. (See figure 1)
2. A 12- by 1-1/4- by 1/8-inch-thick metal straight edge.
3. Graded Standard Sand from Ottawa, Illinois, in accordance with ASTM C-109, preweighed into 300-gram portions.
4. Balance sensitive to 0.1 gram.
5. A No. 6 bristle stencil brush.
6. A pan suitable for transferring sand to individual containers.

Procedure

1. Prepare surface of sample by removing any loose particles such as sand, rubber, etc.
2. Place the plate onto the surface of the sample such that the small rectangular plate rests on the test area, so as to be free from rocking and as steady as possible.
3. Gently pour, from a height of approximately 1-1/2 inch, the previously weighed portion of sand (300 grams) into the hole in the center of the plate so that the entire portion is uniformly distributed and overflowing the hole.
4. Strike off the sand without moving the plate and with as little disturbance as possible. Be careful to retain on the flange of

the plate all of the sand not remaining in the hole. It is recommended that duplicate determinations be made on each sample to insure accuracy.

5. Carefully weigh to the nearest 0.1 gram the sand remaining on the striker plate, and record in column labeled "Wt. Ret. Sand (R)" of worksheet.

6. Steps 2 through 4 should be repeated on a nontextured surface, such as a table top, to obtain standard values.

7. The weight of sand remaining on the striker plate shall be recorded on the bottom of the worksheet as "weight Retained Sand."

#### Calculations

The following formulas are used:

$$X = \frac{D}{W}$$

where: X = Depth of texture per gram of sand

W = Total weight of sand to the nearest 0.1 gram to completely fill the hole in the plate on a flat surface (standard value)

D = Thickness of the plate in inches to the nearest 0.0001 inch

and

$$T = (300 - W - R)X$$

Where: R = Weight of sand to the nearest 0.1 gram remaining on flange of plate on sample whose texture is to be determined.

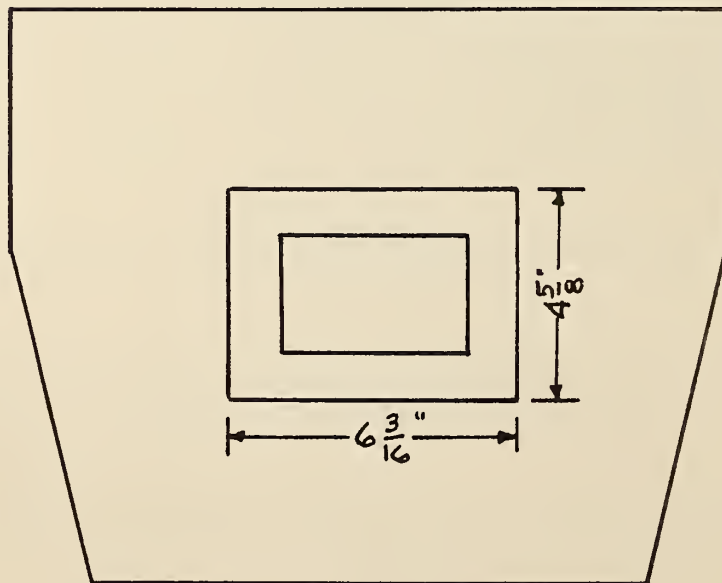
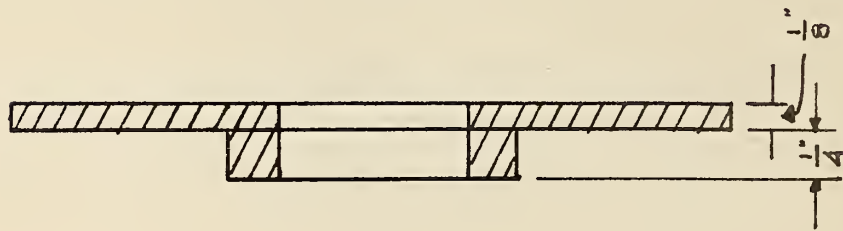
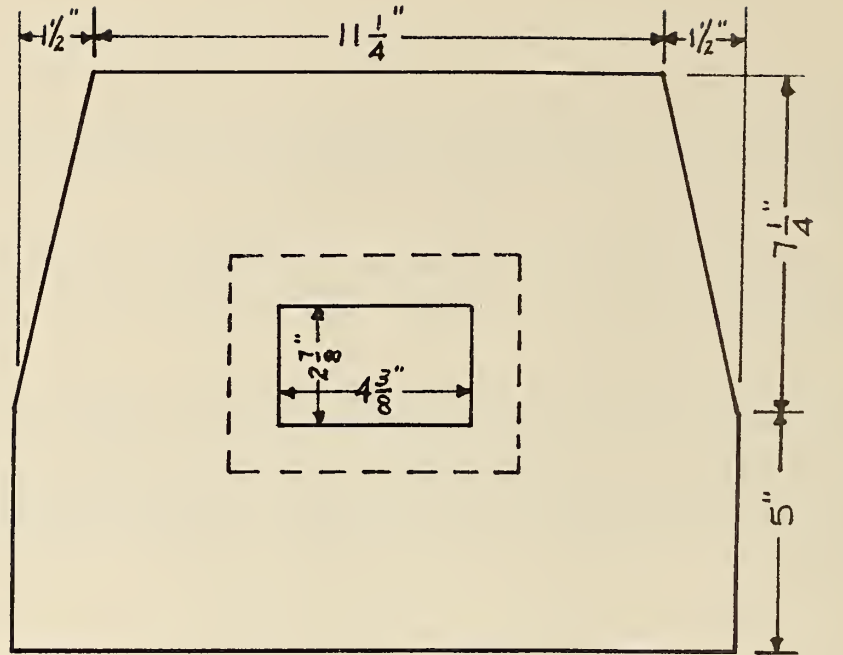
T = depth of texture in inches.

#### Report

Report results to the nearest 0.001 inch.



FIGURE 1



# SAND PATCH DETERMINATION

CONTRACT NO. -

BW 111-012

DATE -

12/12/74

D -

0.637

SAMPLE IDENT. -

COLORADO SAMPLES (A)

REVS. -

1,031,663

X -

0.002841

INITIAL WT SAND -

300 GRAMS

STOP NO. -

5

OPERATOR - J. Mitchell

| SAMPLE NUMBER | TRACK LOCATION | CAN NO.  | WT. RET SAND (g) | AVG. (R) | T     | SAMPLE NUMBER | TRACK LOCATION | CAN NO.  | WT. RET SAND (g) | AVG. (R) | T     |
|---------------|----------------|----------|------------------|----------|-------|---------------|----------------|----------|------------------|----------|-------|
| C16A1         | A              | 5<br>6   | 169.0<br>168.1   | 168.6    | 0.010 | I2N81         | I              | 21<br>22 | 157.6<br>156.7   | 157.2    | 0.042 |
| I1NA2         | B              | 7<br>8   | 146.1<br>147.1   | 146.6    | 0.072 | C26A2         | J              | 23<br>24 | 169.9<br>169.9   | 169.9    | 0.006 |
| C2NA3         | C              | 9<br>10  | 154.4<br>153.0   | 153.7    | 0.052 | I16A3         | K              | 25<br>26 | 166.9<br>168.2   | 167.6    | 0.013 |
| I26B4         | D              | 11<br>12 | 165.9<br>165.1   | 165.5    | 0.018 | I1NA5         | L              | 27<br>28 | 148.0<br>149.4   | 148.7    | 0.066 |
| C26A5         | E              | 13<br>14 | 167.3<br>168.7   | 168.0    | 0.011 | C1NA4         | M              | 29<br>30 | 146.8<br>147.3   | 147.1    | 0.071 |
| I2NA6         | F              | 15<br>16 | 154.6<br>156.2   | 155.4    | 0.047 | C2NA2         | N              | 31<br>32 | 148.5<br>148.1   | 148.3    | 0.067 |
| C1NA7         | G              | 17<br>18 | 153.2<br>152.2   | 152.7    | 0.055 | I26B7         | O              | 33<br>34 | 169.8<br>168.6   | 169.2    | 0.008 |
| I16A8         | H              | 19<br>20 | 163.5<br>164.9   | 164.2    | 0.022 | C16A6         | P              | 35<br>36 | 169.4<br>167.7   | 168.6    | 0.010 |

| CAN NUMBER | WEIGHT RETAINED SAND | WEIGHT TEST SAND | AVERAGE (W) |
|------------|----------------------|------------------|-------------|
| 1          | 171.5                | 128.5            | 128.0       |
| 2          | 172.0                | 128.0            |             |
| 3          | 172.5                | 127.5            |             |
| 4          | 172.0                | 128.0            |             |

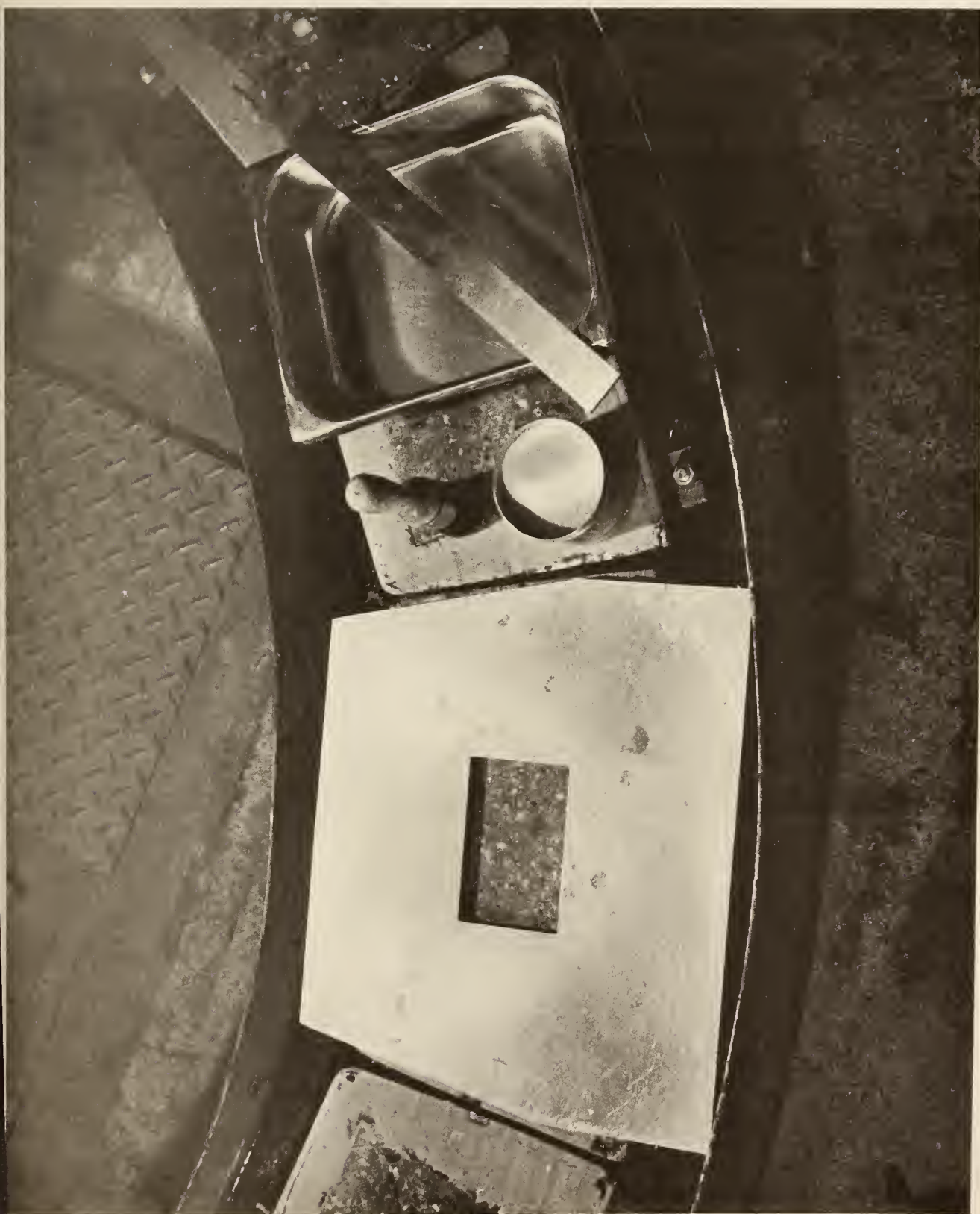
$$X = \frac{D}{W}$$

$$T = (300 - W - R) \times$$

X = Depth of texture per gram of sand  
D = Thickness of plate to 0.0001 inches  
W = Total wt. of sand needed to fill hole in plate on flat surface  
R = wt. of sand remaining on plate  
T = Depth of texture in inches

### APPENDIX A-3

Comparison of Skid Resistance Between a  
Polymerized-Monomer Treated Portland Cement  
Concrete and Untreated Portland Cement  
Concrete as Affected by Laboratory Polishing  
of Three Different Macro-Textured Wearing  
Surfaces



Photograph 1. Sand Patch Equipment





Photograph 2. Sand Patch - Pouring Sand Into Test Area



Photograph 3. Sand Patch - Striking Off Sand





Photograph 4. Sand Patch - Collecting Sand From Flange Of Striker Plate





Photograph 5. Wire Shadow - Photo Equipment





Photograph 6.  
Wire Shadow - Photo - Horizontal Alignment of Light Source





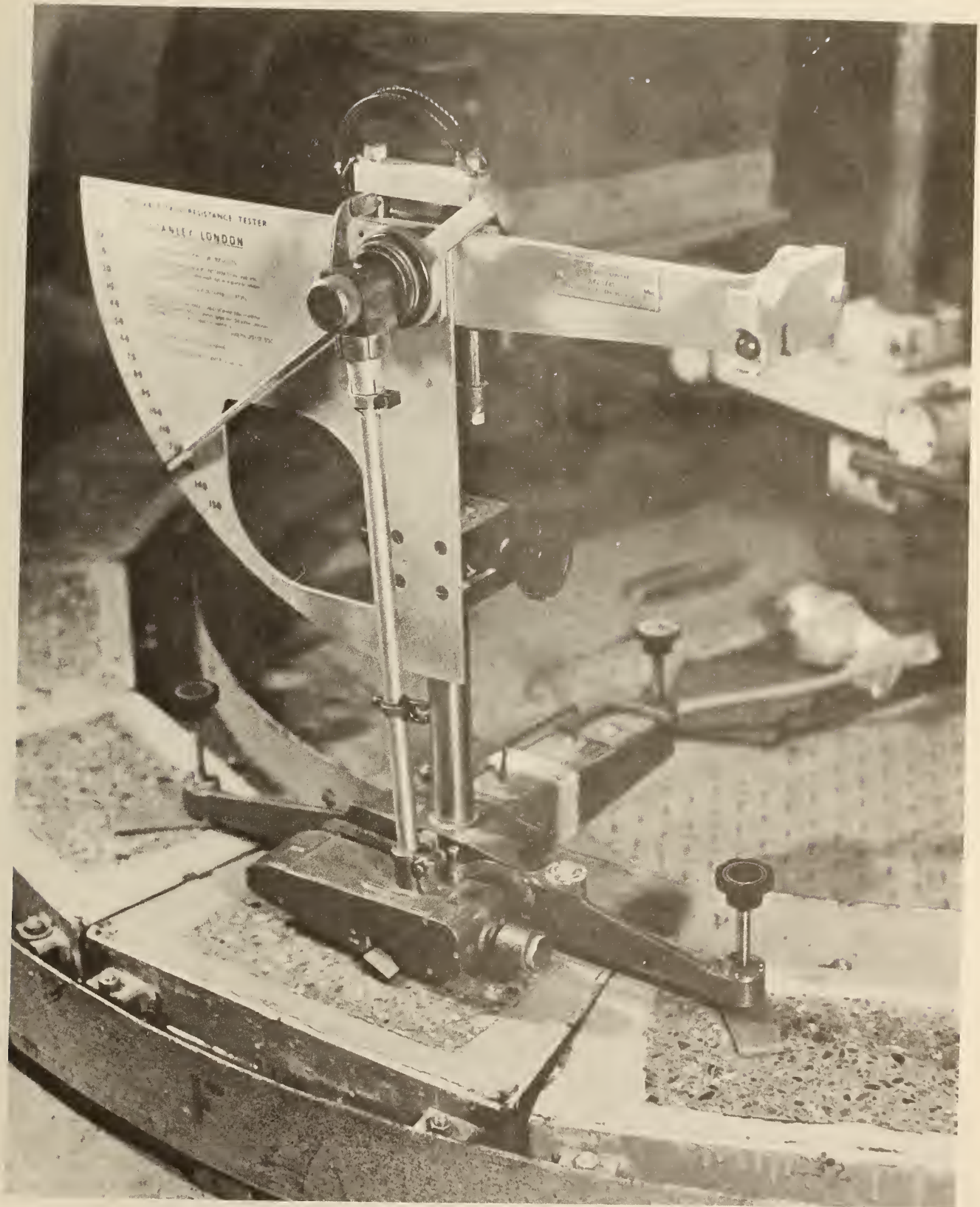
Photograph 7  
Wire Shadow - Photo - Vertical Alignment of Light Source





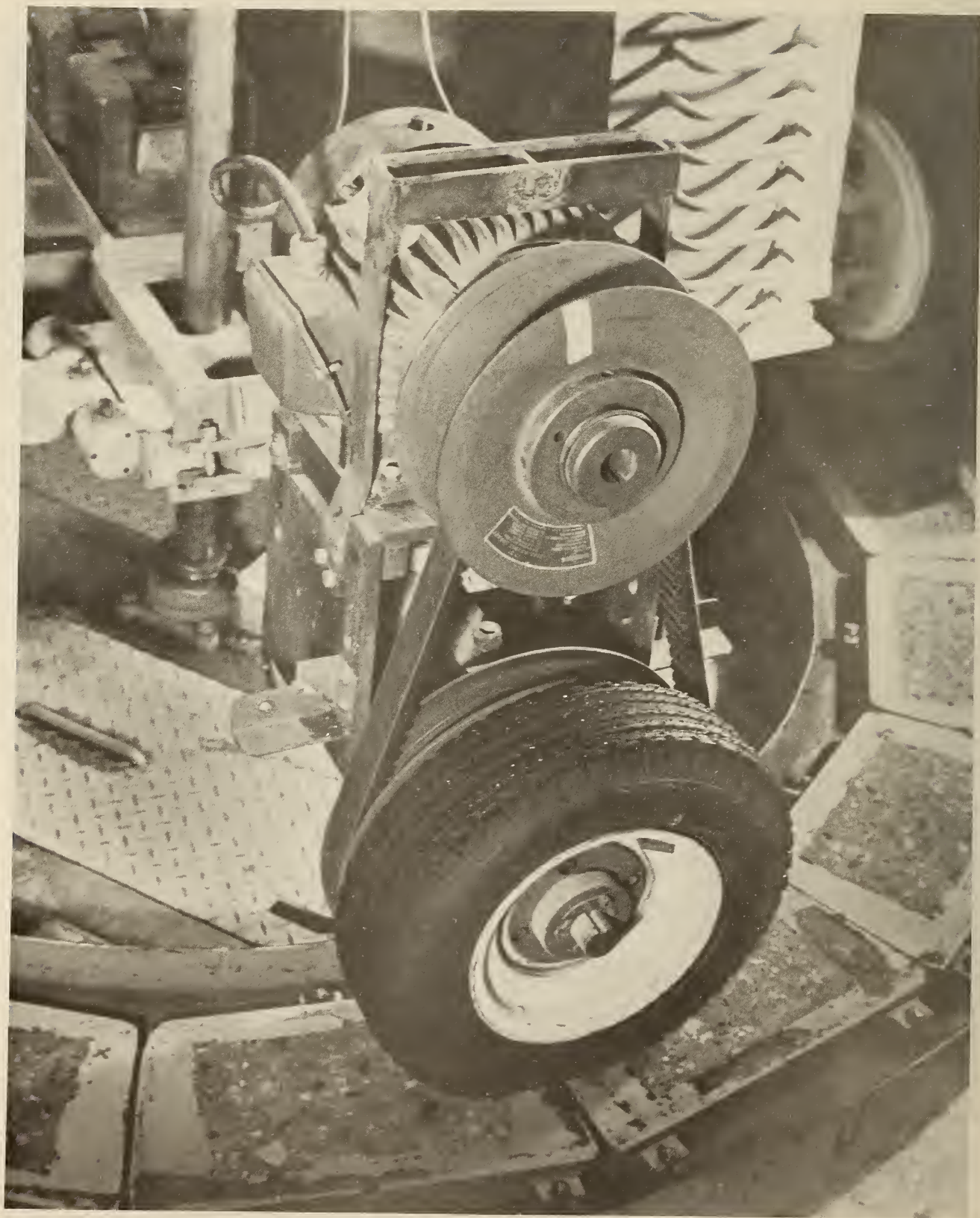
Photograph 8  
Wire Shadow - Photo - Taking Picture



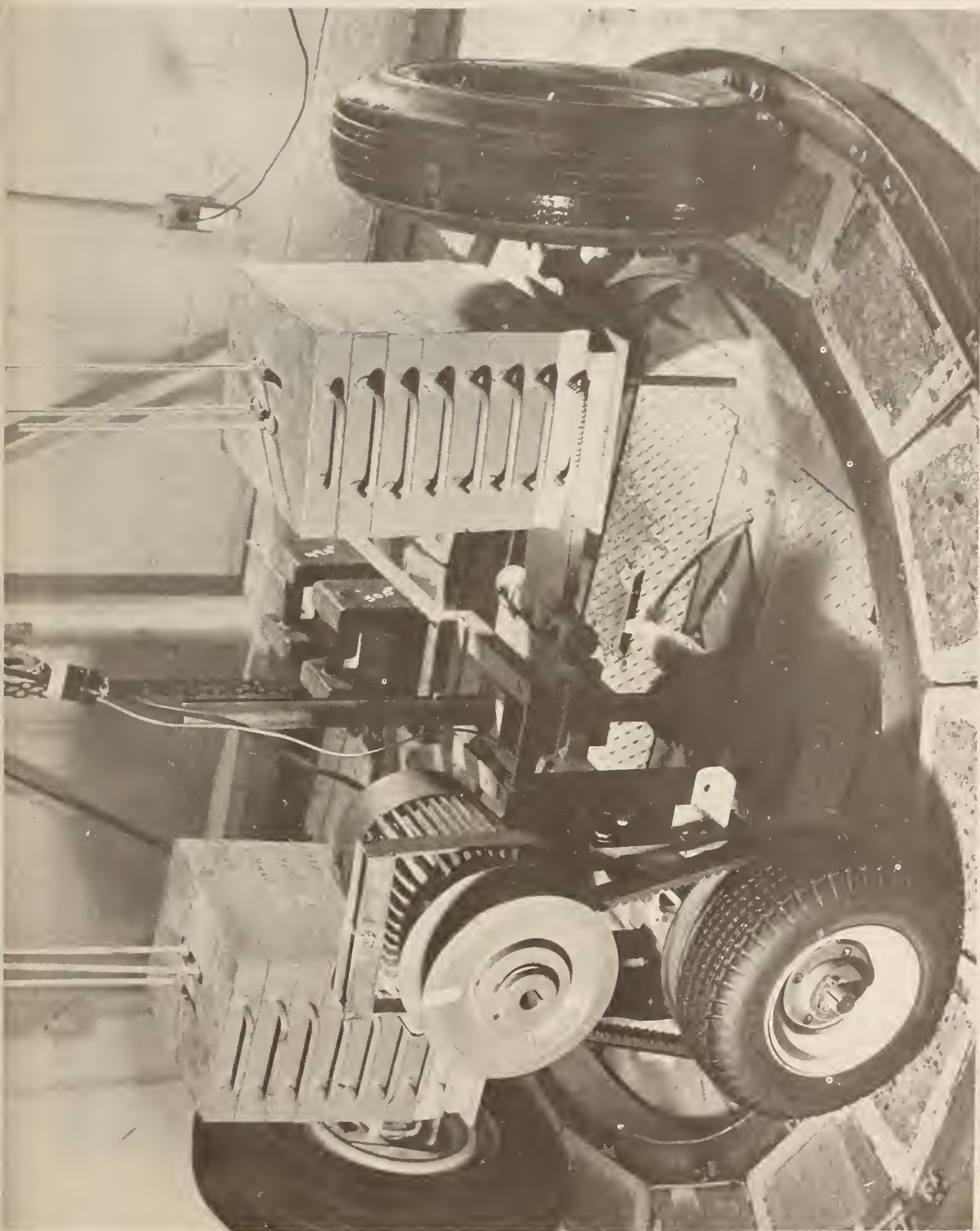


Photograph 9  
BPT Equipment





Photograph 10  
Third Arm Train Equipment



Photograph 11. Third Arm Test Track With Equipment In Position





Photograph 12  
Strain Rate Recorder Printout











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